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A STUDY OF THE SOLID SOLUTION REGIONS IN THE SYSTEM Zr-Te

by

Frances Dolores Jenkins, 1938-

A DISSERTATION

Presented to the Faculty of the Graduate School of the

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In Partial Fulfillment of the Requirements for the Degree

DOCTOR OF PHILOSOPHY

in

CERAMIC ENGINEERING

1972

Advisor

ABSTRACT

Relative intensity measurements of X-ray powder diffraction lines were made of compositions in the region $ZrTe_{0.5}$ to $ZrTe_{2.0}$, fired between 600° and 817°C in evacuated sealed tubes. The experimental relative intensities of samples between ZrTe and $ZrTe_2$ were compared with values calculated for several theoretical models with different Zr vacancy ordering schemes and Te vacancy concentrations. The most probable structural model for compositions in the range ZrTe to $ZrTe_{1.5}$ is one in which the number of Zr vacancies located at z = 0 ranges from 0% to 30%, with the concentration of Te vacancies ranging from 0 to 35%. A structural model of 15% Zr vacancies at z = 0 and Te vacancy concentrations of 0 to 5% is proposed for compositions in the range $ZrTe_{1.5}$ to $ZrTe_2$.

X-ray results indicate a two-phase region between $ZrTe_{0.7}$ and $ZrTe_{1.3}$ in the temperature range from 650° to 817°C. The phases are hexagonal Zr_4Te_3 and a ZrTe-$ZrTe_2$ superlattice. Two-phase material, with a mean composition of $ZrTe_{1.55}$, was observed visually in other samples fired at 600° and 817°C.

All the tellurides reacted with atmospheric moisture at room temperature.

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I. INTRODUCTION

The compounds ZrTe and $ZrTe_2$ have similar crystal structures. ZrTe has the NiAs structure,[1] with hexagonal close-packed layers of non-metal atoms and with metal atoms in all the octahedral sites (Fig. 1). $ZrTe_2$ has the CdI_2 structure,[2] with hexagonal close-packed layers of non-metal atoms with alternate layers of octahedral sites occupied by metal atoms (Fig. 2). It is probable that, in a binary system with both a NiAs-type compound and a CdI_2-type compound, solid solution exists between the compounds.

In the solid solution region, intermediate structures, with increasing metal content, should exhibit increasing site occupancy at the z = 0.5 level. In the Zr-Te system compositions between ZrTe and $ZrTe_2$ should exhibit Zr vacancies, distributed randomly between the z = 0.0 and z = 0.5 levels or exclusively in an ordered manner on the z = 0.5 level.

The purpose of this work was to investigate the structures of intermediate compositions and to determine whether ordering between Zr atoms and vacancies occurs. Information regarding phase relationships, possible structural interpretations, and behavior of the phases in air at room temperature has been obtained.

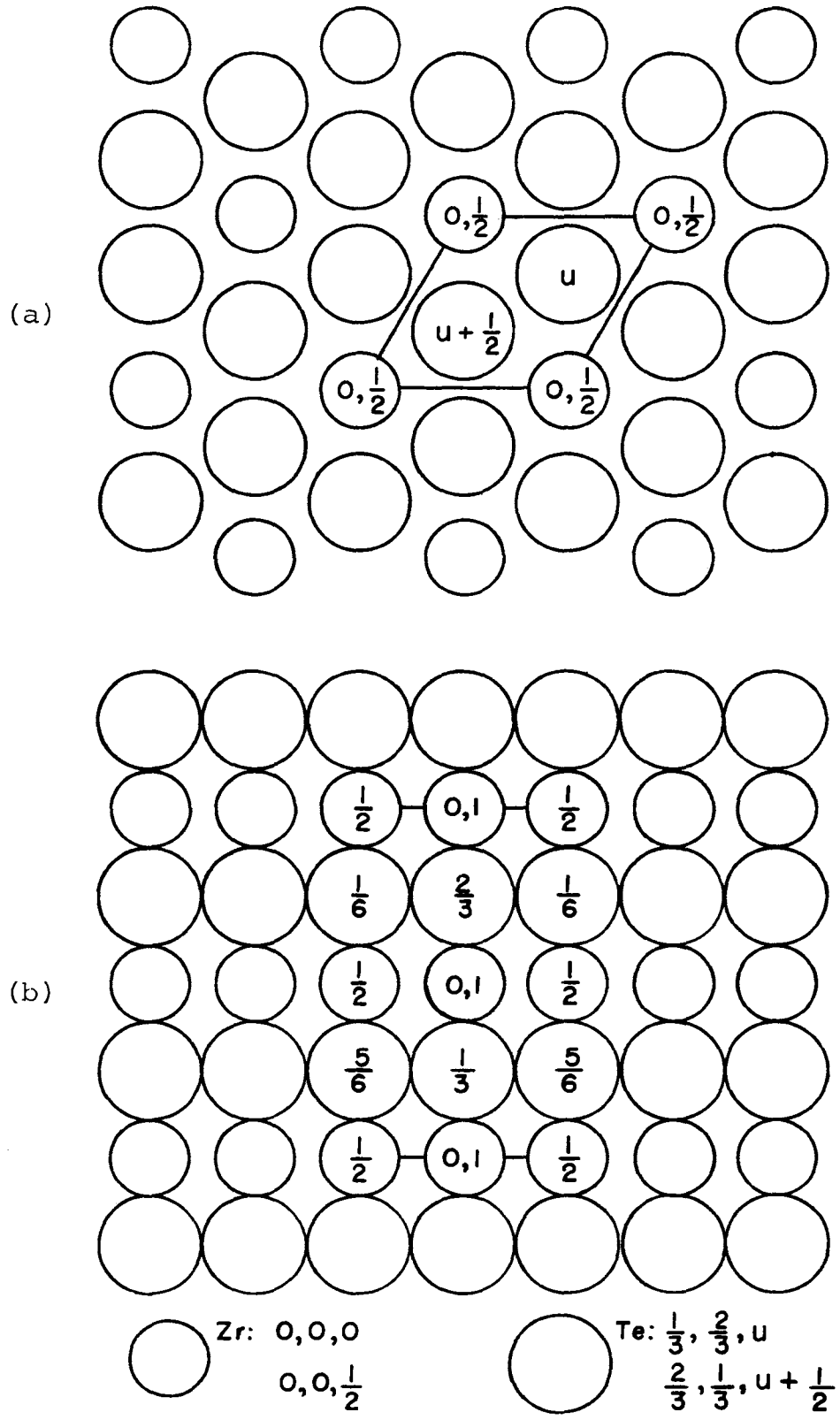

FIGURE 1 The ZrTe structure. (a) (001) projection, (b) ($\bar{1}$10) projection.

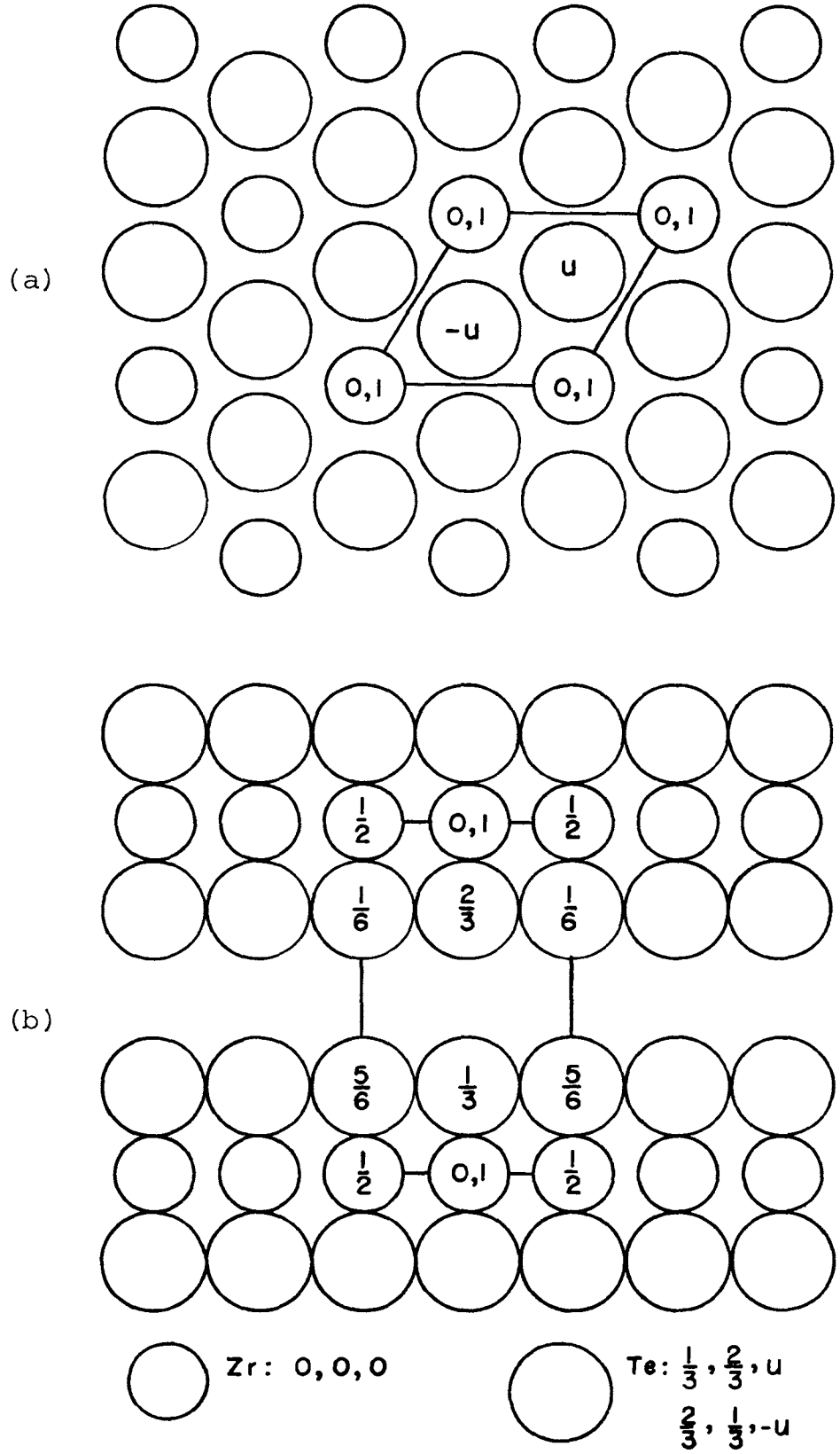

FIGURE 2 The $ZrTe_2$ structure. (a) (001) projection, (b) ($\bar{1}10$) projection.

II. LITERATURE REVIEW

The system Zr-Te has been studied by two groups of workers, Hahn and Ness[3,4] who made a moderately detailed study of the Zr-Te system, and McTaggart and Wadsley,[5] who made a broad survey of group IV metal-chalcogen binary systems.

McTaggart and Wadsley[5] prepared phases at fixed compositions in the binary systems: tri-, di- sesqui-, and monochalcogenides. Their data for the Zr-Te system are summarized in Table I. $ZrTe_3$ was prepared by exposing $\leq$150 mesh Zr metal filings to Te vapor at approximately atmospheric pressure at 600°C for several weeks. The ditelluride was formed by degradation of $ZrTe_3$ at 900°C. This yielded a product of composition $ZrTe_{1.70}$. Zr_2Te_3 was prepared by direct reaction of the elements at 800°C, and it is presumed that ZrTe was also prepared by direct reaction of the elements. All samples were manipulated and stored in a dry argon atmosphere to reduce oxygen pickup.

X-ray powder patterns were obtained by the Debye-Scherrer method, using CuKα radiation. $ZrTe_3$ lattice parameters were obtained from single crystals. $ZrTe_2$ was assigned to the CdI_2 structure on an "obvious" basis; no structure factor calculations were made. On the basis of density measurements, determined pycnometrically, using approximately 5 gram samples with toluene, both the $ZrTe_2$ and Zr_2Te_3 compositions were interpreted in terms of defective anion

TABLE I

Chemical and Crystallographic Data from McTaggart and Wadsley[5]

Compound and Appearance	Composition				Te/Zr Mole Ratio	Density		Crystal System	Unit Cell Dimensions			
	Theor.		Obs.			Obs.	Calc.		a	b	c	γ
	%A	%X	%A	%X								
$ZrTe_3$ Lustrous Black needles	19.24	80.76	19.6	79.3	2.90	6.80	6.82	Monoclinic	5.89	3.93	10.10	98.4°
$ZrTe_2$ Purplish brown	26.33	73.67	29.4	70.5	1.70	6.38	6.36*	Hexagonal	3.952		6.660	
Zr_2Te_3 Black					$\frac{1.90}{1.26}$	6.40	6.40+	"	3.982		6.700	
ZrTe Black powder						6.6	6.48	"	3.95		6.64	

*Calculated for $Zr_{1.12}Te_{1.90}$

+Based on the assumption of a defective anion lattice

lattices. They stated, however, that the measured densities might be low, regardless of the care taken, because of oxidation of the samples. They stated that Zr_2Te_3 could be regarded as only one composition in a solid solution range. ZrTe was assigned to the NiAs structural type.

Chemical analyses were obtained by repeated oxidation-reduction at 700°C in O_2 and H_2 until only the group IV metal oxide remained. They stated that the oxidation-reduction technique was necessary because TeO_2 did not readily sublime.

Hahn and Ness[4] studied the compositional ranges $ZrTe_{0.5}$-$ZrTe_{0.75}$ and $ZrTe_{0.8}$ - $ZrTe_{2.0}$, as well as the $ZrTe_3$ compound. Their starting materials were Zr, leached in HCl, and Te, purified by distillation. Table II summarizes the X-ray data of $ZrTe_2$, ZrTe, and two forms of Zr_4Te_3, obtained by Hahn and Ness. Corrected $\theta/2$ values have been converted to d-values; only lines up to 97° 2θ are shown. Measured lattice parameters of compositions in the range $ZrTe_{0.8}$ - $ZrTe_{2.0}$ are presented in Fig. 3. Fig. 4 shows the variation in density, X-ray and pycnometric, of the same compositions.

Hahn and Ness[3] are in basic agreement with McTaggart and Wadsley[5] on $ZrTe_3$, though the c_o dimension is twice that of McTaggart and Wadsley.[5] The latter group attributed this to twinning in the single crystal investigated by the former.

Hahn and Ness[4] synthesized by heating all samples in the composition range $ZrTe_{0.8}$ - $ZrTe_{2.0}$ in evacuated fused

TABLE II

Diffraction Patterns of Compounds in the Zr-Te System[4]

Zr_4Te_3 (Hexagonal)	Zr_4Te_3 (Tetragonal)	ZrTe (Hexagonal)	$ZrTe_2$ (Hexagonal)
$a=3.76_2 \pm 0.005$Å	$a=3.68_7 \pm 0.005$Å	$a=3.95_3 \pm 0.005$Å	$3.95_0 \pm 0.005$Å
$c=3.86_4 \pm 0.005$Å	$c=9.56 \pm 0.01$Å	$c=6.64_7 \pm 0.005$Å	$6.63_0 \pm 0.005$Å
	4.695 (002) s		
	3.480 (001)*sss		
		3.351 (010) s-	
3.253 (010) m+		3.255 (002) s-	3.255 (002) s
	3.164 (003) m		
		3.046 (011) st	2.998 (011) st
	2.903 (102) st		
	2.60 (110) m		
	2.508 (111) s		
2.484 (011) sst			
	2.392 (103)	2.395 (012) st	2.382 (012) m+
	(004) st		
	2.281 (112) m		
		2.214 (003) ss	2.181 (003) s
	1.998 (104) m	1.997 (110) st	1.979 (110) m

*This is an exact duplication of the data in the paper.

TABLE II (Cont.)

Zr_4Te_3 (Hexagonal)	Zr_4Te_3 (Tetragonal)	ZrTe (Hexagonal)	$ZrTe_2$ (Hexagonal)
1.926 (002) m-			
	1.899 (005) s		
1.881 (110) st			
		1.863 (013) m	1.855 (111)
			(013) m
	1.758 (114) ss		
	1.719 (202) ss		
		1.700 (112) s	1.700 (112) s
1.690 (111) sss	1.689 (105) st		
		1.660 (004)	1.659 (004)
		(021) m	(021) m
1.628 (020) s			
	1.593 (006) s		
	1.558 (212) m		
		1.519 (022) st	1.521 (022) m-
1.499 (021) st		1.492 (014) ss	1.489 (014) s
		1.472 (113) ss	1.472 (113) m-
	1.465 (213)		
	(106) mt		
	1.356 (214) m	1.354 (023) m	1.352 (023) m

TABLE II (Cont.)

Zr_4Te_3 (Hexagonal)	Zr_4Te_3 (Tetragonal)	ZrTe (Hexagonal)	$ZrTe_2$ (Hexagonal)
	1.326 (205) s		
	1.305 (220) s		
1.294 (003) sss			
		1.272 (114)	1.268 (114)
		(121) st	(121) sst
1.244 (022) s	1.248 (215) st		
		1.239 (015) s	1.236 (015) s
	1.206 (206) st	1.206 (122) mt	1.206 (122) m
1.197 (013) m	1.192 (302) ss		1.192 (024) sss
1.172 (121) st	1.167 (310) s		
	1.148 (303) ss		
		1.141 (030) st	1.139 (030) m
	1.134 (312) s		
		1.118 (123) st	1.115 (123) m
	1.096 (313) m		
1.086 (030) st			
	1.078 (225) s	1.079 (032) ss	1.079 (032) ss
	1.048 (314) ss	1.051 (016)	1.049 (016)
		(025) m	(025) st

TABLE II (Cont.)

Zr_4Te_3 (Hexagonal)	Zr_4Te_3 (Tetragonal)	ZrTe (Hexagonal)	$ZrTe_2$ (Hexagonal)
1.039 (122) st			
	1.034 (305) s		

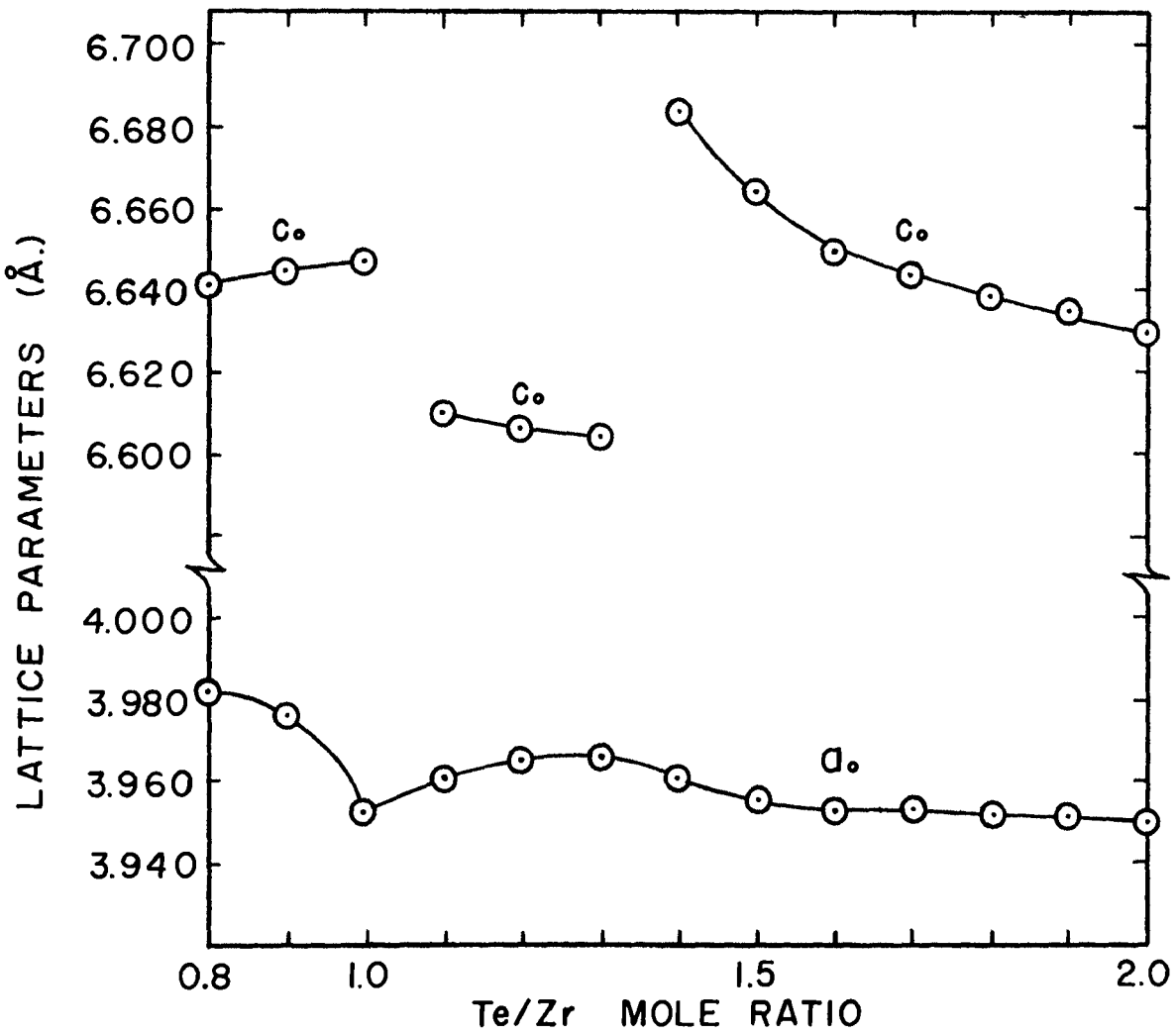

FIGURE 3 The variation in lattice parameters with Te content.

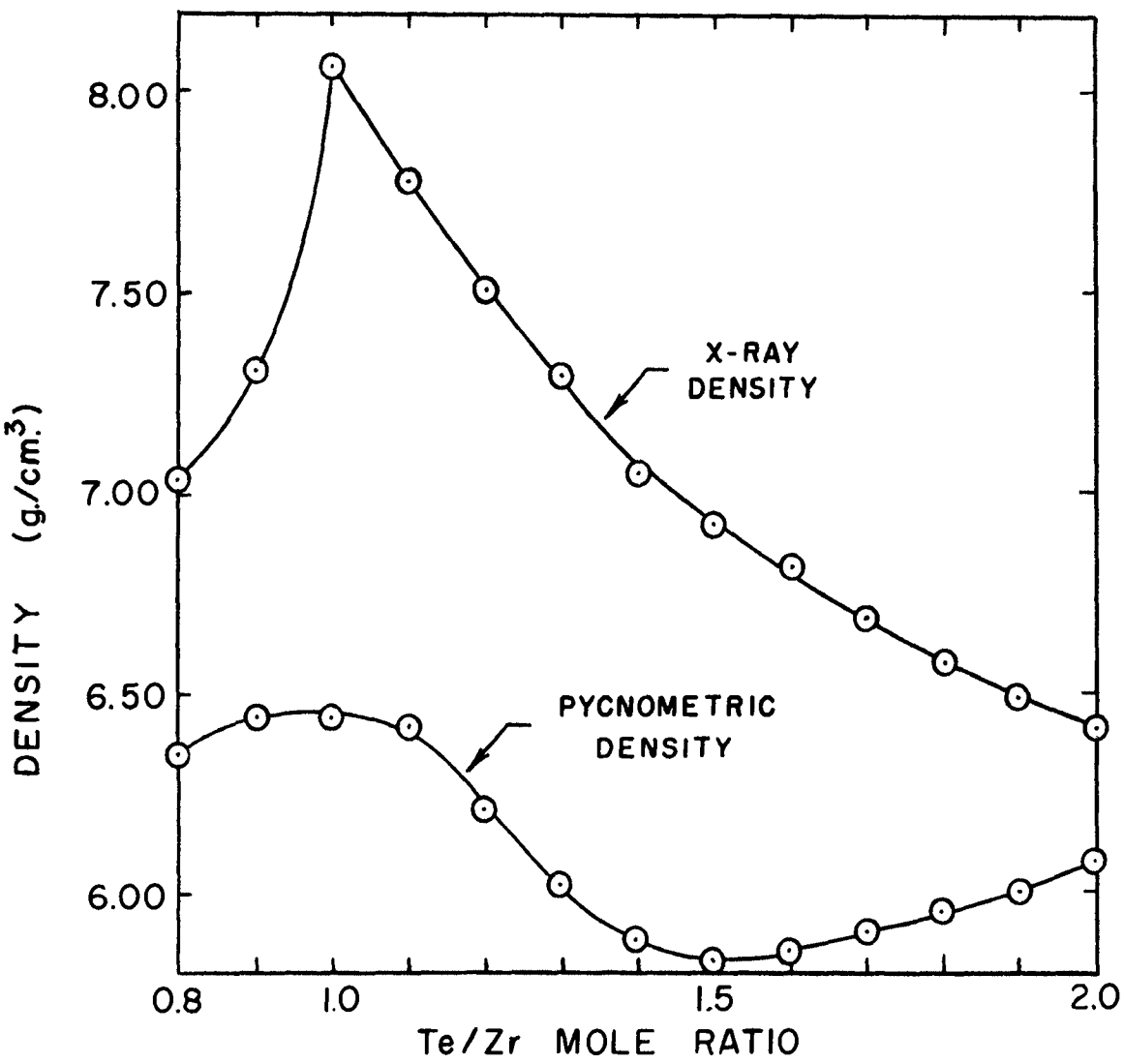

FIGURE 4 The variation in density with Te content.

silica tubes at 850°C for 2 weeks. The compositions with low Te content (the value for "low" was not specified) were fired in Al_2O_3 crucibles in N_2 atmospheres. No statement was made about cooling procedures. Samples with high Te content were said to be brown-violet soft masses; samples with low Te content were gray, metallic, spongy powders. The violet material transformed to a gray color upon exposure to the atmosphere, and the odor of hydrogen telluride was detected. Samples with low Te content were pyrophoric and hence were handled in N_2 atmospheres.

X-ray data of the samples in the region $ZrTe_{0.8}$ to $ZrTe_{2.0}$ were indexed on hexagonal unit cells. Intensity calculations indicated that ZrTe has the NiAs structure and $ZrTe_2$ the CdI_2 structure and that structural continuity is present in the solid solution between the two.

No experimental details were given on the pycnometric density technique, the results of which are shown by Fig. 2. Hahn and Ness[4] stated that "agreement of the X-ray density with that determined pycnometrically shows a proportionately larger defect structure."

Though $ZrTe_2$ single crystals were readily grown in a temperature gradient, ZrTe was considered by Hahn and Ness[4] to be thermally unstable. When ZrTe was fired at 900°C for 4 weeks in a temperature gradient, two crystalline sublimates were obtained at the cooler end, $ZrTe_2$ and a second phase identified as Zr_4Te_3. Single crystal studies showed that Zr_4Te_3 is tetragonal, but the structure was not com-

pletely determined.

The tetragonal phase was not found, however, when Zr_4Te_3 was formed by direct reaction of the elements. Instead, $ZrTe_{0.75}$, as well as $ZrTe_{0.67}$ and $ZrTe_{0.5}$, was reported to be hexagonal, having the WC structure with Zr at 0,0,0, and Te at 1/3,2/3,1/2. The composition range $ZrTe_{0.5}$ - $ZrTe_{0.75}$ was named the Zr_3Te_2 solid solution region, corresponding to similar phase fields in the systems Zr-S and Zr-Se. Lattice parameters and densities of the compositions in the range are summarized in Table III.

Bear and McTaggart[6] attempted to measure and compare rates of oxidation for chalcogenides of Ti and Zr at 150°C and 300°C in air. They were not successful and attributed this to "the samples having different particle size, crystal structure, etc." They observed that the tellurides tended to be more resistant to oxidation than the selenides and sulfides, though not always at the lower temperature.

McTaggart[7] measured resistivities of powders of the group IV metal chalcogenides. The powders were compressed between steel plungers in plastic dies to a constant pressure of 25,000 psi. Resistance was measured either with a Thomson bridge (for resistances below 2Ω) or by direct determination of voltage and current (for resistances above 2Ω). Measurements on pyrophoric samples were made in an argon atmosphere. The results for the Zr-tellurides are summarized in Table IV. All samples exhibited metallic conduction.

TABLE III

Lattice Parameters and Density Values for Compositions in the Zr_3Te_2 Phase Region[4]

	Lattice Parameter		Density	
Composition	a(Å)	C(Å)	X-ray g/cm^3	Pycnometric g/cm^3
$ZrTe_{0.75}$	3.76_2	3.86_4	6.51_3	6.4_1
$ZrTe_{0.67}$	3.76_1	3.85_6	6.12_9	6.5_2
$ZrTe_{0.5}$	3.75_9	3.84_5	5.43_7	5.7_5

TABLE IV

Electrical Properties of Zirconium Telluride Compositions

Composition	Appearance	Specific Resistivity (Ω cm)	Type of Conduction
$ZrTe_{2.90}$	Black lustrous*	0.0004	Metallic
$ZrTe_{1.70}$	Dark purple-brown bronze	0.001	Metallic
$ZrTe_{1.50}$	Black	0.0016	Metallic
$ZrTe_{1.00}$	Black	0.001	Metallic

* Graphitic material

III. EXPERIMENTAL PROCEDURE

A. Sample Preparation

Fine Zr particles were obtained by filing a rod of pure Zr (Special Metals Section, Argonne National Laboratory) with a hardened tool steel file. Care was taken to prevent overheating of the Zr rod. Te was prepared by crushing purified Te sticks (Fisher Scientific Company) with an agate mortar and pestle. To prevent formation of Te fines which could be drawn off during a later evacuation procedure, small quantities of material were crushed at a time.

Thirteen compositions at 0.1 mole Te intervals were prepared in the range $ZrTe_{0.8}$ - $ZrTe_{2.0}$, as well as $ZrTe_{0.5}$, $ZrTe_{0.67}$, and $ZrTe_{0.75}$. Thirty gram batches of each composition were weighed to the nearest 0.0001 gram and placed in small bottles. Samples were tumbled end-over-end by hand for 2 minutes; each composition was then cut into 8 or 16 samples by sample splitting, remixed, and recut into individual samples.

Samples were loaded into 8 mm. i.d. fused silica tubes. The tubes were evacuated, using a mechanical pump, and sealed. The sealed tubes were 10 cm. long.

All samples were fired in an electric muffle furnace. The samples were divided into three groups: 1) $ZrTe_{0.5}$ to $ZrTe_{0.75}$, 2) $ZrTe_{0.8}$ to $ZrTe_{1.4}$, and 3) $ZrTe_{1.5}$ to $ZrTe_{2.0}$. A group of samples was placed on end in a fireclay crucible (about 10 cm high), and the crucible was placed on the floor

of the furnace. The firing temperature was read with a chromel-alumel thermocouple, in contact with the interior base of the crucible. A set-point controller with an on-off relay was used to control furnace temperature. The maximum variation in firing temperature was $\pm$ 3°C.

All samples were fired at 817°C for 130 hours and 600°C for 130 hours. Samples with a Te/Zr mole ratio of 1.4 or lower were also fired at 700°C for 200 hours and 650°C for 500 hours. Samples having a Te/Zr mole ratio of 0.75 or lower were fired at 600°C for 645 hours, with grinding after 145 hours.

Unless otherwise stated, all samples were cooled by quenching in air. The procedure was to remove the fireclay crucible from the furnace and then remove the fused silica tubes from the crucible. This latter step was accomplished within one minute of removal from the furnace. The silica tubes were placed on a slotted insulating brick for cooling at room temperature.

B. X-Ray Techniques

X-ray measurements were made with a General Electric XRD-5 recording diffractometer using CuKα radiation. The following scans were made on all samples: 1) a fast scan (2° 2θ/min.) from 10° to 100° 2θ, 2) a slow scan (0.2° 2θ/min.) from 10° (or 5°) to 100° 2θ, and 3) a counting scan, utilizing a scaler, over the lines observed in the slow scan. Chart speed for all scans was 24 in./hr. Lat-

tice parameters were determined by slow scanning from 40° to 50° 2θ with MgO as the internal standard.

Intensities were determined from the slow scan by cutting and weighing diffraction lines recorded on the chart. Diffraction lines recorded on the chart during the counting scan were also cut and weighed. Relative intensities were calculated using the (011) line as the standard line. In general, relative intensities obtained from the counting and weighing methods agreed within 5%, except when several overlapping peaks were counted. In such cases, distribution of counts among the overlapping peaks was determined from weight measurements, and relative intensities were calculated from scaled counts.

A special sample holder was devised to eliminate misalignment caused by reaction of the sample with atmospheric moisture. A glass slide, with a suitably shaped hole etched through it, was used to hold the material. Front and back of the powder sample were covered with a taut layer of Handiwrap (trademark of Dow Chemical Company), taped at the edges. A glass slide was used to provide a flat surface while the sample was packed from the back and covered, and to provide support for the back of the sample during X-ray scans. The "back-pack" method was chosen to minimize preferred orientation.

Handiwrap exhibits two diffraction maxima, a moderately sharp peak at 21° 2θ and a lower, broader peak at 24° 2θ. Neither interfered with the diffraction patterns of the

tellurides. Saran-Wrap (trademark of Dow Chemical Company) obliterated a large portion of the diffraction pattern and could not be used.

All sample tubes were opened immediately prior to X-ray analysis to minimize hydration.

C. Hydration Studies and Chemical Analysis

Hydration studies were initiated when it became obvious that samples were reacting with the atmosphere during X-ray analysis. When a new sample tube was opened, a small portion of the material was crushed, lightly ground, and poured into a small Al foil crucible, which had previously been weighed. The filled crucible was weighed immediately and stored in dust-free containers. Net sample size was between 0.1 and 0.6 grams; the samples were weighed to the nearest 10 micrograms. The samples were weighed daily until at least 3000 hours had elapsed; X-ray patterns of the samples were then obtained.

Some fully hydrated samples were subsequently chemically analyzed by a roasting technique. The sample was weighed, poured into a small pre-weighed Pt crucible, and reweighed. The crucible was placed in a 600°C furnace for 2-10 hours to oxidize the material. The furnace temperature was then raised to approximately 890°C to complete oxidation and to vaporize the TeO_2.[8] Weight measurements were made prior to raising the temperature to 890°C and during the high temperature soak to follow the progress of the reactions. The

analysis was complete when the sample reached constant weight. The residue from some of the samples was analyzed by X-ray to verify completion of the reaction.

Other procedures were tried before the one cited above was selected. 1) The crucible was held at 150°C in an attempt to remove water. The furnace temperature was then raised to 870°C, and the sample was returned to the furnace for oxidation and subsequent vaporization of the TeO_2. 2) The crucible was placed directly into an 870°C furnace. After 3 or 4 runs, the interior of the Pt crucible exhibited evidence of corrosion, in the form of vertical rippling around the interior sidewall just above the sample, while the exterior of the crucible was unchanged. After 3 or 4 more runs, holes appeared in the crucible wall. This firing procedure was discontinued. 3) The procedure cited in the previous paragraph was used, but the sample was not in the furnace while the temperature was raised to 870°C.

D Reactions with Organic Liquids

Immediately upon opening a tube containing $ZrTe_2$, portions of the fired material were placed in a beaker of toluene and in one of pyridine. The beakers were covered and placed at room temperature in a fume hood for 10 days. Liquid level was maintained 2 cm. above the material.

After 10 days the samples were removed from the liquid, ground, and analyzed by X-ray. The sample soaked in toluene was dry-ground, while the sample soaked in pyridine was wet-ground.

E. Computer Techniques

A computer program was written to calculate relative intensities of diffraction lines for different values of the Te coordinate, u, for the $ZrTe_2$ structure. It also was used to calculate relative intensities for varying degrees of Zr vacancy disorder and Te vacancy concentration in compositions between ZrTe and $ZrTe_2$. Atomic scattering factors were computed from numerical Hartree-Fock wave functions.[9]

A program for calculation of corrected d-spacings and 2θ values from lattice parameters was also written. All computer programs are presented in Appendix A.

IV. RESULTS AND DISCUSSION

A. Appearance of Fired Samples

The fired material varied in color from a gold-brown at $ZrTe_2$ to black or blackish brown at ZrTe. The texture was that of a porous powder sinter, easily crushed and soft during grinding.

Some samples fired at 600° and 817°C consisted of two phases. A hard globular material, with metallic, copper luster, was present in the center of the sample. The material was more difficult to crush and grind than the surrounding powder. Chemical analyses of the two-phase samples yielded average compositions near $ZrTe_{1.55}$. The materials are discussed more fully in Section F.

Material fired at 600°C was generally darker brown in color than that fired at 817°C. However, a more striking difference between the fused silica sample tubes fired at 600°C and those fired at 817°C was noted. Sample tubes heated at 600°C were clear, while many heated to 817°C had a black interior coating. The coating commonly flaked off when the sample tubes were opened. Unfortunately, there was an insufficient amount of material for X-ray analysis. The appearance of the samples and the sample tubes is summarized with the X-ray data in Appendices B through I.

There was no reaction between the material and the fused silica tubes, even with the low Te compositions, at any temperature. Firing samples of low Te content in Al_2O_3

crucibles, as done by Hahn and Ness,[4] appears unnecessary. Even when there was a black coating on the wall of the sample tubes, high-Te samples were a gold-brown in color. The presence of the black coating did not affect the sample color or X-ray pattern in any way.

Black coating did not form in all cases with high-Te samples, but were formed in all cases with samples of $ZrTe_{1.2}$ or lower Te content. Black coating was formed in sample tubes fired as low as 650°C.

There are three possible explanations for the coating. The first is that the black coating indicates the presence of residual water vapor in the sample tube after evacuation. The vapor would react or be absorbed by some of the telluride and produce the coating. The justification for this reasoning is that the X-ray samples turned black during analysis, and all samples turned black during hydration in air. Samples turned black within 12 hours of exposure, in some cases within 1 hour. A second possibility is that water vapor was released from the silica tubing at 650°C and higher, turning telluride on the tube walls black. A third possibility is that the black color indicates formation of a second phase of considerably lower Te content (not the metallic material discussed above). X-ray patterns of samples removed from blackened tubes exhibited lines other than those attributable to known telluride phases. The hydration studies showed that reaction between components for compositions of low Te content was incomplete at 600°C. Thus, if

the second phase was not formed at 600°C, the silica vials would remain clear at this firing temperature.

B. Chemical Analyses

Chemical analysis was achieved by roasting to oxidize the telluride and subsequently vaporizing the TeO_2, leaving ZrO_2. The equations used to calculate the analyzed Te/Zr ratio from the weight of the residual ZrO_2 are shown in Appendix J. The results of the chemical analyses, along with observations on the samples, are summarized in Table V. The column headed "Free Zr" indicates the presence of free or partially reacted Zr in the sample. With the exception of a few samples marked "ℓ", all samples were primarily black powder. The columns headed "Time Elapsed" indicate total heating time from the start of the analysis procedure.

Comparison of the first two columns, which give the intended and analyzed values of the Te/Zr mole ratio, respectively, shows that mixing was far from ideal. In retrospect, it can be seen that the sample splitting technique does not work well when two different types of particulate matter are to be mixed, namely filings which can intertwine themselves and a granular powder which is free-flowing. Far less error would have been introduced if the samples had been individually weighed and mixed.

Some observations on the oxidation behavior of these Zr-tellurides can be made. In general, those samples having an analyzed Te/Zr mole ratio of 1.3 or less did not form a

TABLE V

Observations on Chemical Analysis Samples

Sample	Analyzed Te/Zr Mole Ratio	Free Zr	600°C Firing				870°-890°C Firing	
			Time Elapsed, Hours	High Temp. Color	Low Temp. Color	Surface Crust Formed	Time Elapsed, Hours	Color
2.0-4[a]	2.12	No	9.6	Lt. gray	Lt. gray	Yes	48.1	Beige
1.9-6	1.86	No	12.6	Lt. gray	Lt. gray	Yes	62.5	Beige
1.8-4	1.91	No	4.8	White	White	Yes	41.6	Beige
1.7-4s	1.65	No	5.8	Lt. gray	Lt. gray	Yes	42.6	Cream
1.7-4ℓ	1.47	No	10.6	Yellow, Lt. gray	White, Lt. gray	No	38.8	Cream
1.6-6s	1.69	No	5.8	Gray	Gray	Yes	44.7	Cream
1.6-6ℓ	1.30	No	6.0	Yellow, Gray	White, Gray	Yes	24.6	Beige
1.5-5ℓ[b]	1.39	No	4.1	Lt. gray	Lt. gray	Yes	29.1	Cream
0.8-7	0.67	Yes	12.4	Yellow, Gray	White, Gray	No	25.3	Cream
0.9-7	0.94	Yes	6.0	Yellow, Gray	White, Gray	No	26.4	Cream
1.0-8	1.33	Yes	5.1	Yellow, Gray	White, Gray	No	34.5	Cream

[a] intended Te/Zr mole ratio is 2.0; sample number is 4.

[b] Sample 1.5-5s was dropped.

TABLE V (Cont.)

Sample	Analyzed Te/Zr Mole Ratio	Free Zr	600°C Firing				870°-890°C Firing	
			Time Elapsed, Hours	High Temp. Color	Low Temp. Color	Surface Crust Formed	Time Elapsed, Hours	Color
1.1-7	1.29	Yes	10.6	Yellow	White	No	35.9	Cream
1.2-10	1.14	Yes	10.5	Yellow, Gray	White, Gray	No	45.6	Cream
1.3-7	1.37	Yes	10.7	Yellow, Gray	White, Gray	No	47.0	Cream
1.4-7	1.35	No	5.2	Yellow, Gray	White, Gray	No	47.1	Cream
1.5-1ℓ	1.53	No	4.50	Dk. gray	Dk. gray	Yes	33.8	Cream
1.5-1s	1.62	No	11.2	Gray, Yellow	Gray	--	23.8	Cream
1.6-4	1.67	No	1.6	White	White	Yes	29.73	Cream
1.7-6	1.19	Yes	1.8	White	White	Yes	52.46	Cream
1.8-6	1.43	No	3.6	White	White	--	51.9	Cream
1.9-4	1.70	No	8.1	White	White	--	45.2	Cream
2.0-6	2.02	No	4.9	White	White	Yes	48.9	Cream
0.50-2	0.21	Yes	5.0	White, Cream	White, Cream	No	20.9	Cream
0.67-2	0.69	Yes	2.3	Yellow	White	No	44.5	Cream
0.75-2	0.83	No	14.8	Yellow	White	No	62.9	Cream

TABLE V (Cont.)

Sample	Analyzed Te/Zr Mole Ratio	Free Zr	600°C Firing				870°-890°C Firing	
			Time Elapsed, Hours	High Temp. Color	Low Temp. Color	Surface Crust Formed	Time Elapsed, Hours	Color
0.9-1	0.82	No	11.1	Lt. gray	Lt. gray	Yes[c]	36.1	Cream
1.9-5	1.62	No	3.9	White	White	Yes	57.2	Cream
1.8-5	2.13	No	10.9	White	White	Yes	59.5	Cream
1.6-8ℓ	0.99	No	2.6	Lt. gray	Lt. gray	Yes	28.9	White
0.8-6	0.87	Yes	1.6	Yellow	White	No	24.5	White
0.9-6	1.03	Yes	2.0	Yellow	White	No	23.3	Cream
1.0-6	0.91	Yes	1.7	Yellow	White	No	23.2	White
1.1-6	1.02	Yes	1.7	Yellow	White	Yes	45.7	White
1.2-6	1.29	Yes	2.9	Yellow	White	Yes	33.3	Cream
1.3-6	1.42	Yes	3.0	Lt. gray	Lt. gray	Yes	48.6	Cream
1.4-6	1.96	No	3.5	White	White	Yes	47.2	Cream
1.4-5	1.51	No	3.8	White, Gray	White, Gray	Yes	45.4	White
1.3-5	1.15	Yes	3.6	Yellow, Gray	White, Gray	--	44.0	White
1.2-5	0.98	Yes	3.2	Yellow	White	No	53.4	White
1.1-5	1.22	--	2.0	White	White	--	105.4	White
0.9-4	0.82	No	1.9	Gray	Gray	--	21.6	White

[c] Sample oxidized under surface crust as well.

TABLE V (Cont.)

Sample	Analyzed Te/Zr Mole Ratio	Free Zr	600°C Firing: Time Elapsed, Hours	600°C Firing: High Temp. Color	600°C Firing: Low Temp. Color	600°C Firing: Surface Crust Formed	870°-890°C Firing: Time Elapsed, Hours	870°-890°C Firing: Color
0.9-2	0.92	--	2.0	Gray	Gray	No	22.9	White
1.1-4	0.62	Yes	2.0	Lt. gray	Lt. gray	No	21.7	White
1.1-2	1.05	No	4.1	Lt. gray	Lt. gray	Yes	46.7	White
0.8-4[d]	0.84	No	--	--	--	--	25.3	Cream
0.8-2	0.72	Yes	--	--	--	--	21.6	Cream
1.0-4	1.32	--	--	--	--	--	31.8	Cream
1.0-2	1.11	--	--	--	--	--	--	--
1.3-4[e]	--	--	--	--	--	--	--	--
1.3-2	0.78	--	--	--	--	--	20.1	Cream
1.4-4	0.95	--	--	--	--	--	25.0	Cream
1.4-2	1.52	--	--	--	--	--	22.9	Cream
1.2-4	0.68	No	--	--	--	--	23.8	Cream
1.2-2	0.82	--	--	--	--	--	22.5	Cream

[d] These four samples had a preliminary heating at 150°C before being placed in an 870°C furnace.

[e] This sample was lost, because the Pt crucible developed large holes. All samples in this group were placed directly into an 870°C furnace.

surface crust during the 600°C preheat. The material remained moderately free-flowing and was almost completely oxidized, i.e., white at room temperature. Some exceptions were noted, the 0.9-1 sample, which had been exposed to the atmosphere for 8351 hours, an unusually long exposure. This sample formed a surface crust and was light gray in color instead of white after the 600°C preheat. (Other samples were open to the atmosphere 500 hours at the most, and many of them for much less time.) For samples which had an analyzed Te/Zr mole ratio larger than 1.3, the time spent at 600°C was insufficient to oxidize the material completely. This was due primarily to the formation of a surface crust of oxidized material. This crust apparently prevented rapid penetration of oxygen into the sample, as indicated by the fact that material underneath the crust was dark gray in color. Thus, the oxide or oxides which formed on the materials of high Te content sintered more readily and slowed the rate of oxidation of the entire sample.

It is believed that the material observed to be yellow at high temperatures was $ZrTe_3O_8$, the only intermediate phase stable in air in the system ZrO_2-TeO_2.[8] No X-ray patterns were made of this material.

This writer does not agree with the comment of McTaggart and Wadsley[5] regarding the difficulty of removing TeO_2 by vaporization. TeO_2 was readily vaporized at 870°C. The time required to reach constant weight on the high Te content samples was not considered excessive.

C. Relative Intensity Calculations and Measurements

The theoretical intensity of the diffraction line from any (hkl) plane is calculated by multiplying the following factors: multiplicity of the (hkl) plane, the temperature factor, the Lorentz-polarization factor, and the square of the structure factor.[10]

For crystal structures with centers of symmetry, such as the CdI_2- and NiAs-type structures, the structure factor, F, is calculated from the equation

$$F = \sum_N f_N \cos 2\pi(hx_N + ky_N + lz_N),$$

where N = number of atoms in the unit cell,

f_N = atomic scattering factor for the Nth atom,

h,k,l = Miller indices of the plane, and

x_N, y_N, z_N = coordinates of the Nth atom in the unit cell.

If a diffractometer is used, no correction for absorption of X-rays by the sample is required.

It is seen from the equation that the intensity of a diffraction line is dependent on the coordinates of the atoms in the unit cell. However, the effect of vacancies in a crystal on the intensity of a diffraction line is also readily apparent. Assume a crystal composed of A and B atoms, each atom having a specific site in the crystal. Assume also that vacancies are introduced onto the A lattice, while the B lattice remains intact, without any change in

crystal structure. If 10% of the A lattice sites are vacant, i.e., 90% of the A lattice sites are occupied, the atomic scattering factor for the A atom must be multiplied by 0.9, the fraction of filled A sites. This will change the value of the calculated intensity of any line. Introducing vacancies onto specific lattice sites requires a proportional decrease in the corresponding atomic scattering factor, resulting in changes in the calculated intensity values. Intensity calculations of both types, i.e., for different values of atomic coordinates and for various amounts of vacancies on both the Zr and Te sublattices, have been made in this study.

In the experimental work, the (011) line was generally the most intense. This line was chosen, therefore, as the standard line for all compositions in the range $ZrTe_{0.8}$ - $ZrTe_{2.0}$, and all relative intensity values were calculated with respect to it. This permits comparison of relative intensities across the phase region.

In some cases the (001) and (002) lines, as well as other lines, split, indicating distortion of the structure. The new line had the higher d-spacing and was much smaller. These are identified as "a" and "b" in the Appendices. Because it was difficult to divide these peaks accurately, the combined intensity of the "a" and "b" peaks was used. This distortion was not observed in the early work. It is not known whether this was the result of a lack of distortion in the samples or to continued improvement in the per-

formance of the X-ray equipment as experimental work proceeded.

The results of the X-ray studies are tabulated in Appendices B through I. They are grouped according to firing procedures and chemical analysis results. The values tabulated are uncorrected d-spacings and relative intensities.

In the $ZrTe_2$ structure a Te layer is bonded to only one Zr layer, whereas in the ZrTe structure it is bonded to two Zr layers. One would expect the Te layer in the ZrTe structure to be situated midway between the two Zr layers. In the $ZrTe_2$ structure, however, the spacing between the Zr layer and adjacent Te layers, expressed by the parameter u in the coordinates of the Te atoms (see Fig. 2), could be some value other than $\pm$ 1/4, the value of u in ZrTe.

Because the intensity of a diffraction line is dependent on the coordinates of all the atoms in the unit cell, the relative intensities of diffraction lines will vary if one of the atomic coordinates, such as u, is changed. It is possible, therefore, to determine the value of u, the spacing between adjacent Zr and Te layers in $ZrTe_2$, by comparing experimental values of relative intensities with corresponding theoretical values, calculated for different assumed values of u.

A computer program was written to calculate theoretical relative intensities, I_{hkl}/I_{011}, for the $ZrTe_2$ structure with varying values of u. The temperature factor was not incorporated. The results are shown in Figs. 5 and 6.

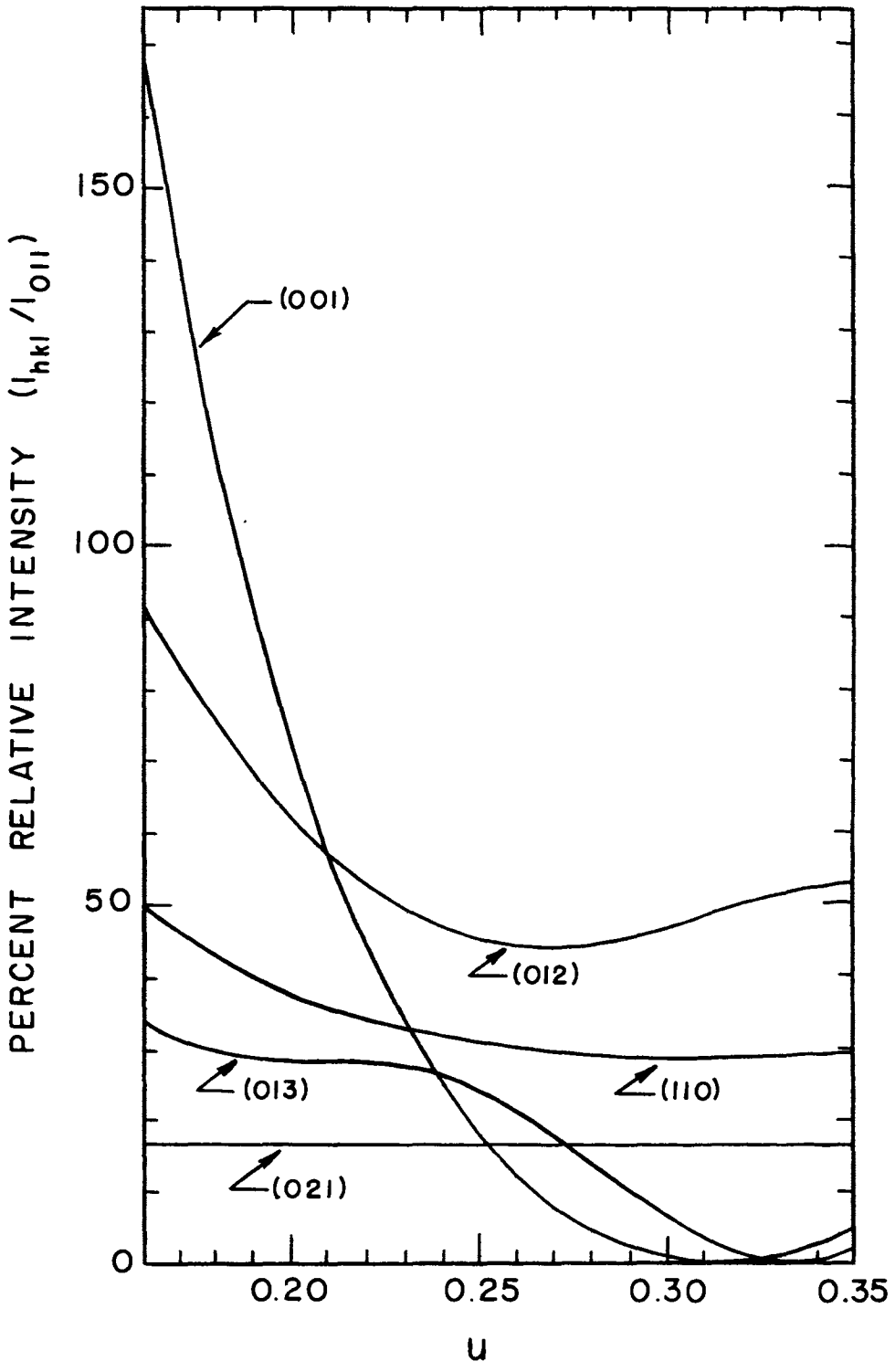

FIGURE 5 Theoretical values of I_{hkl}/I_{011} for values of u, the Te atom coordinate, in the $ZrTe_2$ structure.

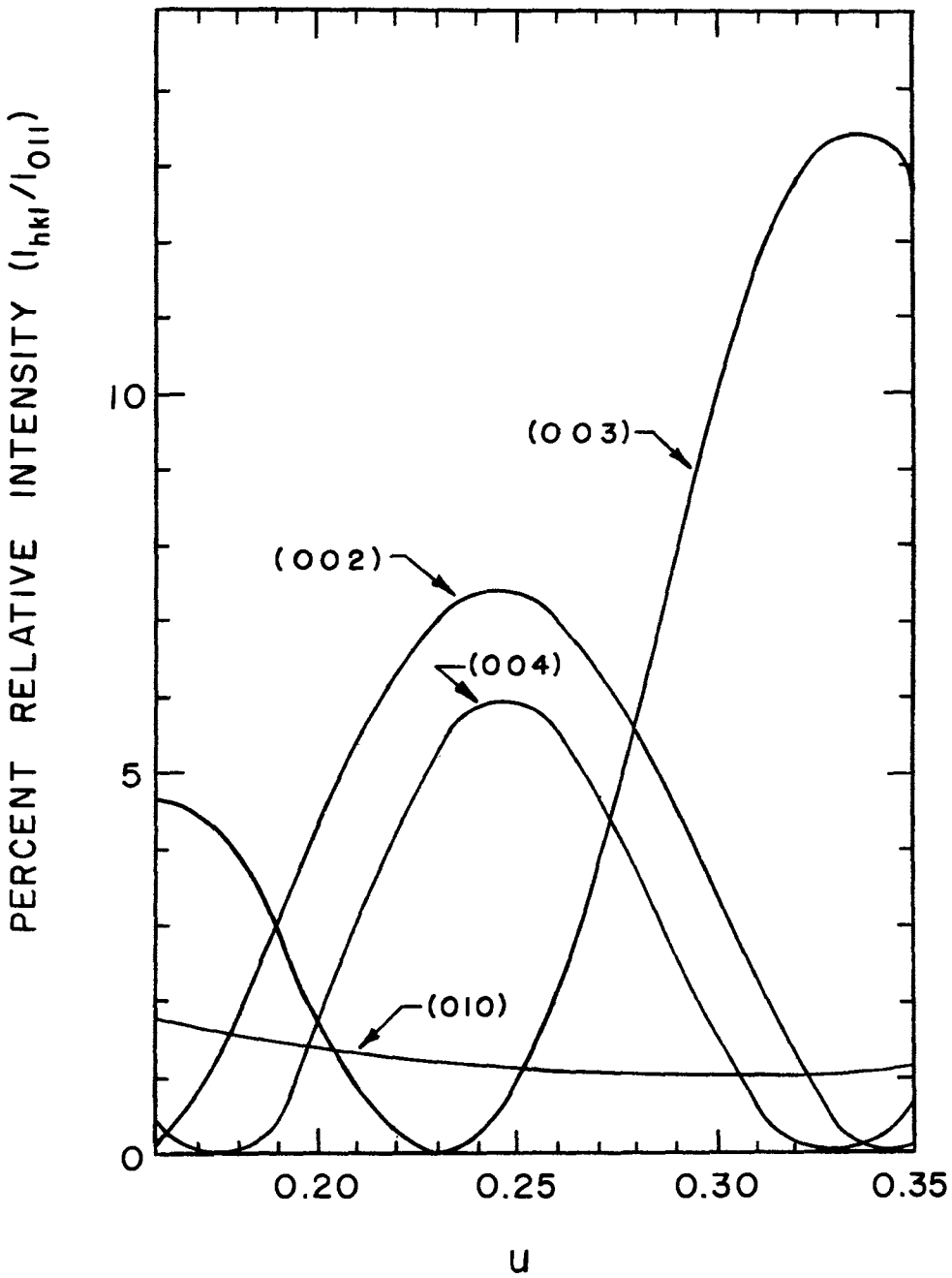

FIGURE 6 Theoretical values of I_{hkl}/I_{011} for values of u, the Te atom coordinate, in the $ZrTe_2$ structure.

Experimental relative intensities for most of the lines are shown in Table VI.

There is considerable scatter in the results presented in Table VI. Of these samples, only 2.0-4 and 2.0-6 have been chemically analyzed. The results of these samples should be given more weight, because both are known to be ditellurides. Agreement between the two sets of relative intensity values is good.

Consider the relative intensity values of only these latter two samples. The experimental value for the (001) line (55%) indicates a value of 0.21 for u on Fig. 5. However, the experimental values for the (002) and (003) lines (40% and 12%, respectively) are much higher than the respective values (5% and 1%) calculated theoretically, shown in Fig. 6, for u = 0.21. The experimental relative intensity values for the (110) and (012) line (28% and 45%) suggest a value of 0.26 or 0.27 for u. With this value of u, the experimental results for the (001), (002), and (003) lines (55%, 40%, and 12%) are much higher than the respective theoretical values ($\sim$9%, $\sim$7%, and $\sim$3%). This indicates that preferred orientation affected the intensities. Preferred orientation of these samples is to be expected, because $ZrTe_2$ is a layer structure with easy cleavage between Te-Te layers.

The effect of preferred orientation can be removed by considering relative intensities of successive orders of lines of a given type, e.g., the basal pinacoids. The com-

TABLE VI

Experimental Values of I_{hkl}/I_{011} for $ZrTe_2$ Samples

(hkl)	Sample No. and Firing Temperature				
	2.0-1 817°C	2.0-2 817°C	2.0-3 600°C	2.0-4 817°C	2.0-6 600°C
(001)	121%	114%	15%	55%	58%
(002)	190	267	12	40	47
(003)	54	41	4	12	14
(110)	28	36	27	29	27
(012)	70	55	40	44	46
(013)	26	38	18	25	27
(021)	52	50	22	25	31

puter program was modified to calculate the following relative intensities: $I_{00\ell}/I_{002}$ and I_{0k0}/I_{110}. The results for $I_{00\ell}/I_{002}$ as a function of u are shown in Fig. 7. The values I_{010}/I_{110} and I_{030}/I_{110} were constants, 3.7 and 7.8%, respectively. The corresponding experimental relative intensities are shown in Table VII.

Again, we assign greater weight to the 2.0-4 and 2.0-6 samples. The experimental values for the (001) and (003) lines (∿130% and 34%) indicate a value of 0.26 to 0.27 for u. No decision on the value of u can be made from the experimental value of I_{030}/I_{110} (∿18%), because it is higher than the value calculated theoretically (7.8%). This possibly indicates that the x and y atomic coordinates of the Zr and Te atoms are slightly different from those assumed in the model, the CdI_2 structure. Theoretical relative intensity calculations could be made for different x- and y-coordinates of the atoms. Such calculations were not undertaken in this thesis.

As stated previously, compositions between ZrTe and $ZrTe_2$ will have vacant Zr lattice sites in a ZrTe model lattice. The concentration of Zr vacancies in these intermediate compositions is most easily determined by converting the composition based on a Te/Zr mole ratio into an "equivalent composition" based on a Zr_2Te_2 lattice. Table VIII summarizes the "equivalent composition" and concentration of Zr vacancies, in terms of mole fraction, for the composition range ZrTe to $ZrTe_2$. This involves the assumption that

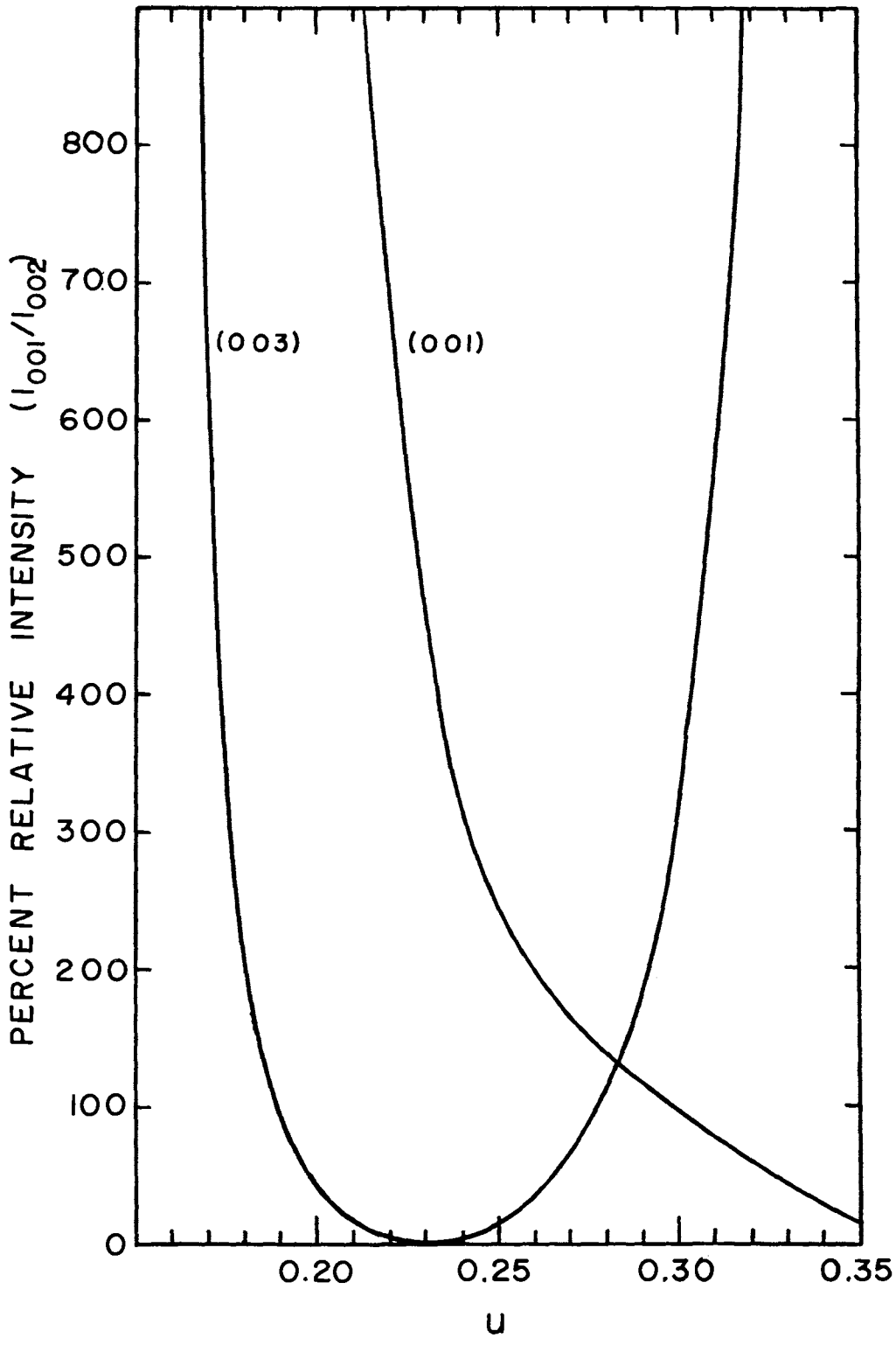

FIGURE 7 Theoretical values of $I_{00\ell}/I_{002}$ for values of u, the Te atom coordinate, in the $ZrTe_2$ structure.

TABLE VII

Experimental Values of $I_{00\ell}/I_{002}$ For $ZrTe_2$ Samples

00ℓ	Sample Number and Firing Temperature				
	2.0-1 817°C	2.0-2 817°C	2.0-3 600°C	2.0-4 817°C	2.0-6 600°C
(001)	64%	43%	125%	126%	141%
(003)	28	15	33	34	34

TABLE VIII

Mole Fraction of Zr Vacancies For Some Zr-Telluride Compositions, Based on a ZrTe Lattice With No Te Vacancies

Composition	Equivalent Composition	Zr Vacancies, Mole Fraction
$ZrTe_{1.0}$	$Zr_{2.00}Te_{2.0}$	0.00
$ZrTe_{1.1}$	$Zr_{1.82}Te_{2.0}$	0.18
$ZrTe_{1.2}$	$Zr_{1.67}Te_{2.0}$	0.33
$ZrTe_{1.3}$	$Zr_{1.54}Te_{2.0}$	0.46
$ZrTe_{1.4}$	$Zr_{1.43}Te_{2.0}$	0.57
$ZrTe_{1.5}$	$Zr_{1.33}Te_{2.0}$	0.67
$ZrTe_{1.6}$	$Zr_{1.25}Te_{2.0}$	0.75
$ZrTe_{1.7}$	$Zr_{1.18}Te_{2.0}$	0.82
$ZrTe_{1.8}$	$Zr_{1.11}Te_{2.0}$	0.89
$ZrTe_{1.9}$	$Zr_{1.05}Te_{2.0}$	0.95
$ZrTe_{2.0}$	$Zr_{1.00}Te_{2.0}$	1.00

there are no defects in the Te lattice.

Zr vacancies can be randomly distributed between both Zr layers in the model lattice, clustered in one layer, or partially ordered on both layers. Earlier discussion showed how vacancies can change the calculated intensity values of diffraction lines. Likewise, for a given composition calculated intensities will vary with the degree or ordering, or concentration, of Zr vacancies on the two lattice sites.

A computer program was written to calculate relative intensities, I_{hkl}/I_{011}, for different degrees of Zr vacancy ordering in the composition range ZrTe - $ZrTe_2$. A disorder parameter expresses the distribution of Zr vacancies on the Zr lattice sites. When the disorder parameter has a value of 0.0, the Zr vacancies are confined to the (002) plane, or, in terms of equivalent positions, to the 0,0,1/2 Zr site. This is the completely ordered condition. When the disorder parameter has a value of 0.2, 20% of the Zr vacancies are on the (001) plane, or, in terms of equivalent position, 20% of the Zr vacancies are on the 0,0,0 site and 80% on the 0,0,1/2 site. This is a partially ordered state. When the disorder parameter has a value of 0.5, the Zr vacancies are distributed equally on both layers, or on both Zr sites, the disordered condition. The distribution of Zr vacancies on Zr lattice sites is summarized in Table IX.

Results of the theoretical calculations for three lines, (001), (012), and (110), are shown in Figs. 8 to 10, respectively. The value of the Zr disorder parameter is

TABLE IX

Distribution of Zr Vacancies on Zr Lattice Sites for Values of the Disorder Parameter

	Distribution of Zr Vacancies	
Zr Disorder Parameter	Zr Lattice Sites (0,0,0)	(0,0,½)
0.0	0%	100%
0.1	10%	90%
0.2	20%	80%
0.3	30%	70%
0.4	40%	60%
0.5	50%	50%

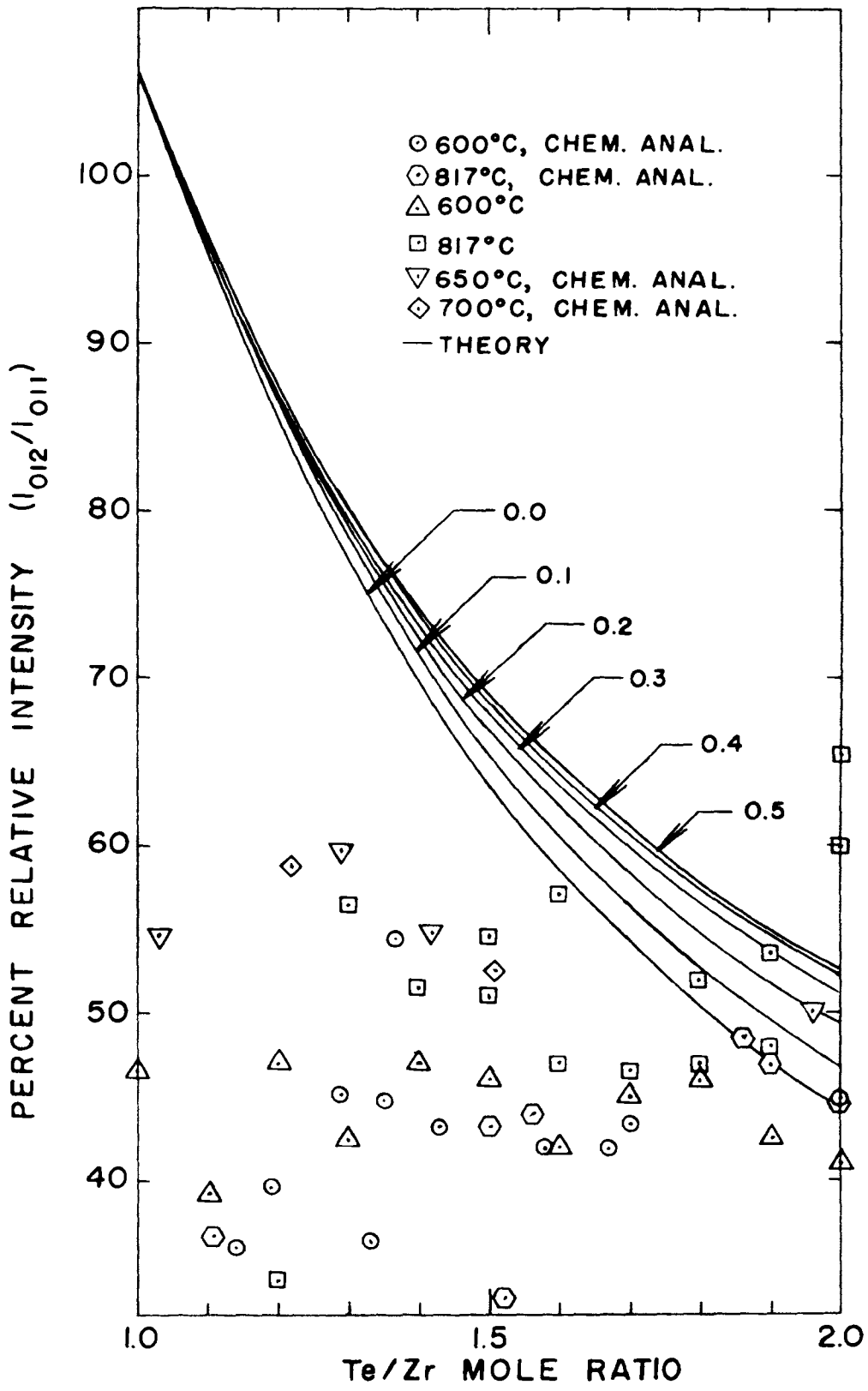

FIGURE 8 Theoretical and experimental values of I_{012}/I_{011} for different values of Zr disorder parameter.

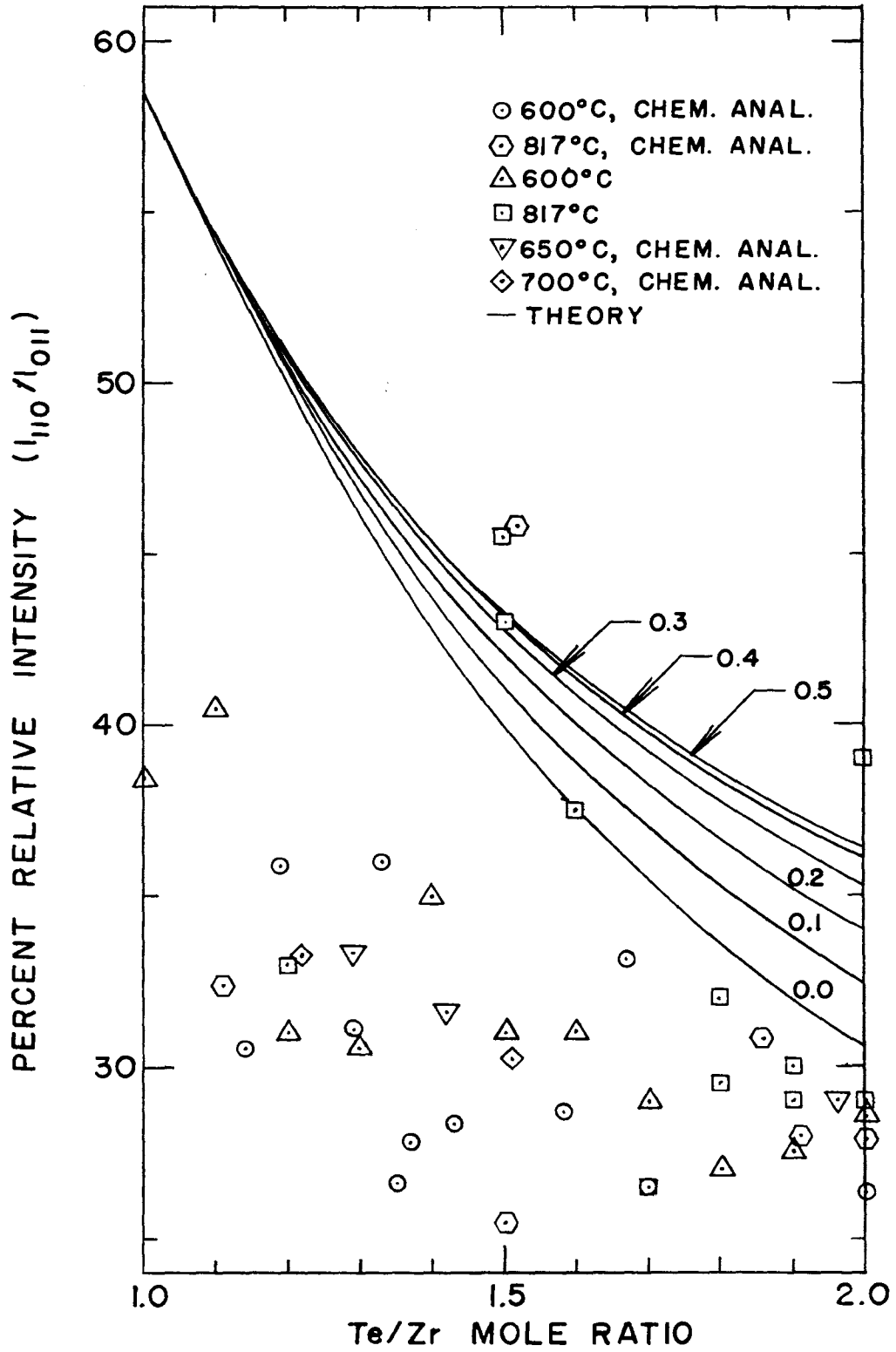

FIGURE 9 Theoretical and experimental values of I_{110}/I_{011} for different values of Zr disorder parameter.

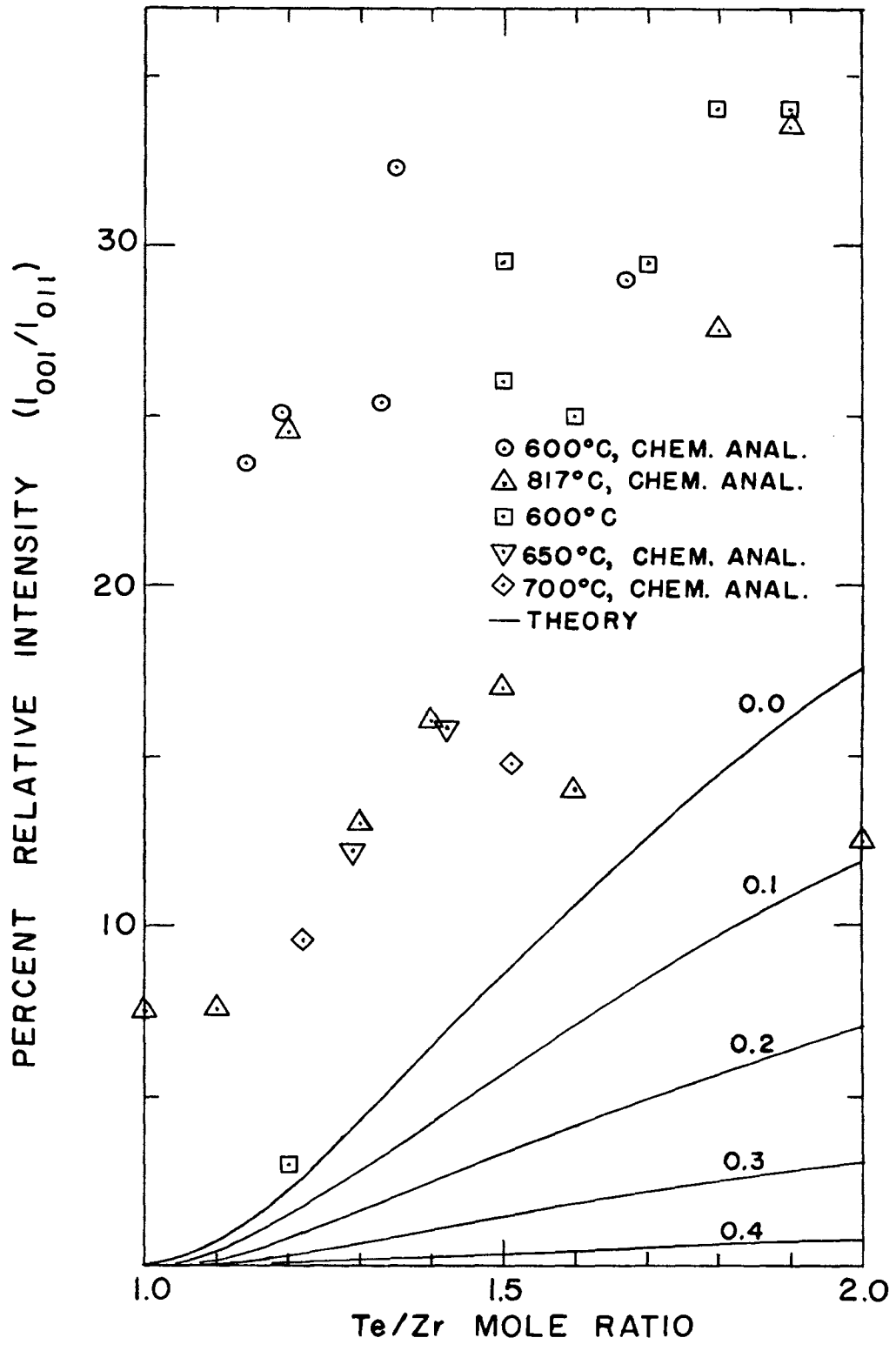

FIGURE 10 Theoretical and experimental values of I_{001}/I_{011} for different values of Zr disorder parameter.

indicated for each curve. The experimental relative intensities are also shown. These experimental values are means of the relative intensities obtained by the weighing method (slow scan) and the counting method. For a few samples, there was a large difference (up to 17%) between relative intensities obtained by the two methods, the value from weighing being higher than that from counting. However, to be consistent in presentation of the data, the average of these widely differing values was used. In addition to differences in relative intensities between the two scans, the intensities of all the peaks in the count scan were lower than those in the slow scan, which was run first.

Figs. 8 and 9 show that the theoretical relative intensities of the (012) and (110) lines increase with decreasing Te content and, for a given composition, increase with increasing disorder in the Zr lattice. On the other hand, the theoretical relative intensity of the (001) line (Fig. 10) decreases with decreasing Te content and increasing Zr disorder. For all three lines, very few experimental results lie near the theoretically calculated values. It should be noted that theoretically the (012) peak becomes the most intense peak in ZrTe. This was never observed experimentally.

The experimental data for the (001) line indicate probable preferred orientation in the samples, because they lie considerably above the theoretically calculated values. The experimental data for the (110) line also verifies this,

being lower than expected theoretically; the (011) line would be enhanced by preferred orientation, thereby reducing the value of I_{110}/I_{011}.

In the earlier discussion on $ZrTe_2$, it was said that by considering relative intensities of lines of a given type, such as the basal pinacoids, the effect of preferred orientation in the sample could be removed. Accordingly, the relative intensity values, I_{001}/I_{002}, were calculated from the theoretical and experimental results in the composition range $ZrTe_{1.0}$ to $ZrTe_{2.0}$. The results of these calculations and the experimental values are shown in Fig. 11. The theoretical calculations show a decrease in relative intensity, I_{001}/I_{002}, with decreasing Te content and with increasing disorder of the vacancies in the Zr lattice. Most of the experimental results lie within the region bounded by the theoretical values. For compositions with a Te/Zr mole ratio of 1.4 or higher, the experimental results indicate some disordering of vacancies in the Zr lattice. Remembering that a value of 0.5 for the Zr disorder parameter expresses the completely disordered state, it can be seen that the X-ray samples have a fairly high degree of ordering of the Zr vacancies. The experimental relative intensity values for compositions having a Te/Zr mole ratio of 1.3 or less lie, in general, outside the region bounded by the theoretical values.

All of the above mentioned theoretical values were calculated on the assumption that the Te lattice was free

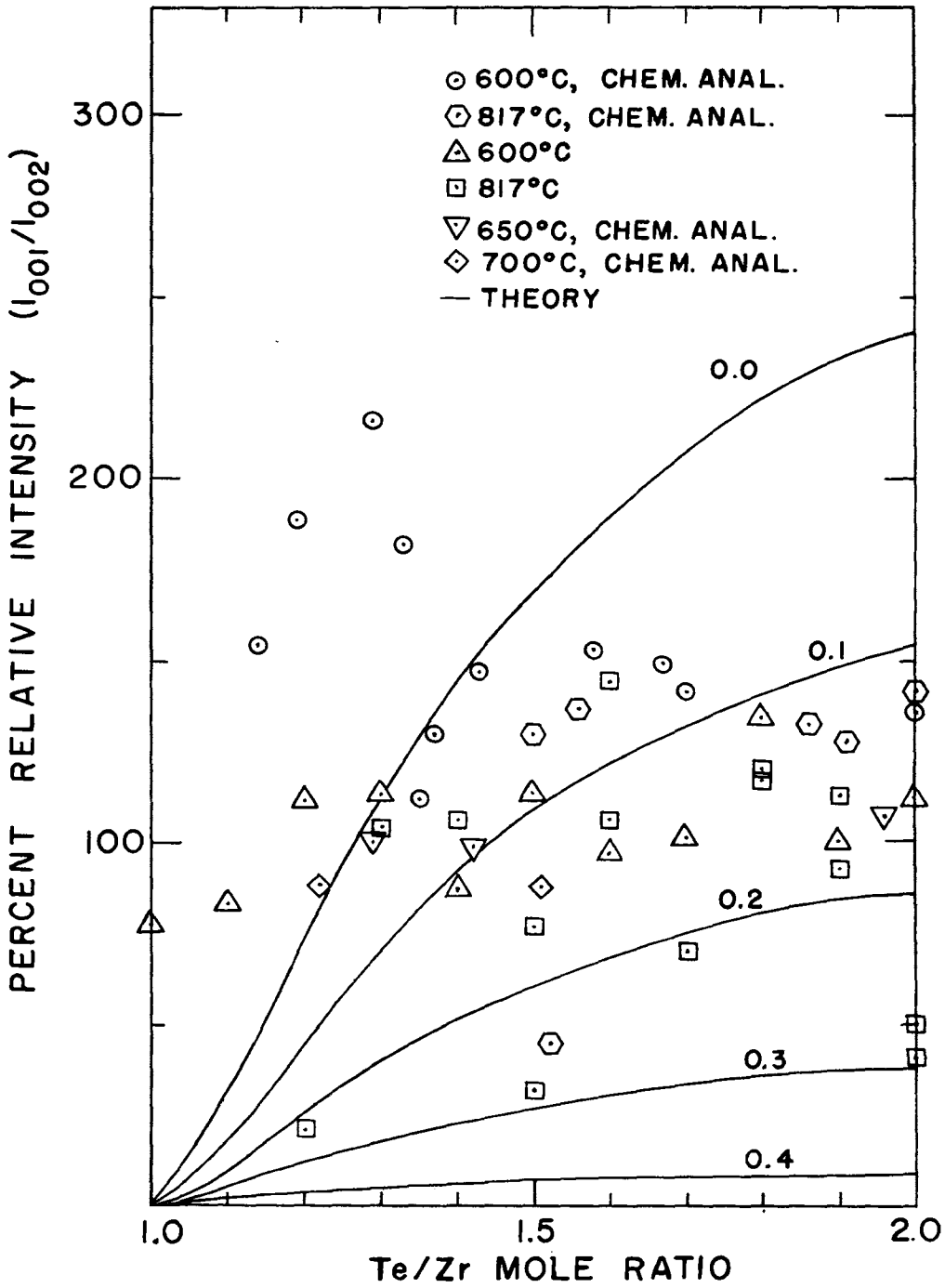

FIGURE 11 Theoretical and experimental values of I_{001}/I_{002} for different values of Zr disorder parameter.

of defects and that all defects were on the Zr lattice. The lack of agreement between some of the experimental points and the theoretical relative intensity values in Fig. 11 suggests that for these points the model of a defect-free Te lattice is not correct. The lack of agreement between theoretical and experimental values of I_{012}/I_{011} and I_{110}/I_{011} could be the result of an incorrect vacancy model, rather than solely by preferred orientation in the sample. It is informative, therefore, to determine how vacancies on the Te lattice, in addition to the vacancies inherent on the Zr lattice by virtue of the composition, affect the theoretical relative intensities. The computer program for calculating relative intensities was modified to perform these calculations. The program is included in Appendix A.

When Te vacancies are introduced, a proportional number of vacancies must be substituted for Zr atoms in the Zr lattice to maintain a constant composition. These vacancies are <u>in addition</u> to those already present on the Zr lattice by virtue of the composition. The concentration of Zr vacancies as a function of Te vacancy concentration is again most easily determined by converting the composition based on a Te/Zr mole ratio into an "equivalent composition" based on a Zr_2Te_2 lattice. This "equivalent composition" is summarized in Table X as a function of Te vacancy concentration for compositions in the range ZrTe - $ZrTe_2$.

The concentration of Zr vacancies as a function of Te

TABLE X

"Equivalent Compositions" For Te Vacancy Concentrations

Composition Te/Zr Mole Ratio	Percentages of Vacant Te Sites		
	0%	10%	20%
$ZrTe_{1.0}$	$Zr_{2.00}Te_{2.0}$	$Zr_{1.800}Te_{1.80}$	$Zr_{1.600}Te_{1.60}$
$ZrTe_{1.1}$	$Zr_{1.82}Te_{2.0}$	$Zr_{1.636}Te_{1.80}$	$Zr_{1.454}Te_{1.60}$
$ZrTe_{1.2}$	$Zr_{1.67}Te_{2.0}$	$Zr_{1.500}Te_{1.80}$	$Zr_{1.333}Te_{1.60}$
$ZrTe_{1.3}$	$Zr_{1.54}Te_{2.0}$	$Zr_{1.386}Te_{1.80}$	$Zr_{1.231}Te_{1.60}$
$ZrTe_{1.4}$	$Zr_{1.43}Te_{2.0}$	$Zr_{1.286}Te_{1.80}$	$Zr_{1.142}Te_{1.60}$
$ZrTe_{1.5}$	$Zr_{1.33}Te_{2.0}$	$Zr_{1.200}Te_{1.80}$	$Zr_{1.068}Te_{1.60}$
$ZrTe_{1.6}$	$Zr_{1.25}Te_{2.0}$	$Zr_{1.125}Te_{1.80}$	$Zr_{1.000}Te_{1.60}$
$ZrTe_{1.7}$	$Zr_{1.18}Te_{2.0}$	$Zr_{1.059}Te_{1.80}$	$Zr_{0.941}Te_{1.60}$
$ZrTe_{1.8}$	$Zr_{1.11}Te_{2.0}$	$Zr_{1.000}Te_{1.80}$	$Zr_{0.889}Te_{1.60}$
$ZrTe_{1.9}$	$Zr_{1.05}Te_{2.0}$	$Zr_{0.947}Te_{1.80}$	$Zr_{0.842}Te_{1.60}$
$ZrTe_{2.0}$	$Zr_{1.00}Te_{2.0}$	$Zr_{0.900}Te_{1.80}$	$Zr_{0.800}Te_{1.60}$

TABLE X (Cont.)

Composition Te/Zr Mole Ratio	Percentages of Vacant Te Sites		
	30%	40%	50%
$ZrTe_{1.0}$	$Zr_{1.400}Te_{1.40}$	$Zr_{1.200}Te_{1.20}$	$Zr_{1.000}Te_{1.00}$
$ZrTe_{1.1}$	$Zr_{1.272}Te_{1.40}$	$Zr_{1.091}Te_{1.20}$	$Zr_{0.909}Te_{1.00}$
$ZrTe_{1.2}$	$Zr_{1.167}Te_{1.40}$	$Zr_{1.000}Te_{1.20}$	$Zr_{0.834}Te_{1.00}$
$ZrTe_{1.3}$	$Zr_{1.078}Te_{1.40}$	$Zr_{0.924}Te_{1.20}$	$Zr_{0.769}Te_{1.00}$
$ZrTe_{1.4}$	$Zr_{1.000}Te_{1.40}$	$Zr_{0.857}Te_{1.20}$	$Zr_{0.714}Te_{1.00}$
$ZrTe_{1.5}$	$Zr_{0.933}Te_{1.40}$	$Zr_{0.800}Te_{1.20}$	$Zr_{0.666}Te_{1.00}$
$ZrTe_{1.6}$	$Zr_{0.874}Te_{1.40}$	$Zr_{0.749}Te_{1.20}$	$Zr_{0.624}Te_{1.00}$
$ZrTe_{1.7}$	$Zr_{0.824}Te_{1.40}$	$Zr_{0.706}Te_{1.20}$	$Zr_{0.588}Te_{1.00}$
$ZrTe_{1.8}$	$Zr_{0.778}Te_{1.40}$	$Zr_{0.666}Te_{1.20}$	$Zr_{0.555}Te_{1.00}$
$ZrTe_{1.9}$	$Zr_{0.736}Te_{1.40}$	$Zr_{0.631}Te_{1.20}$	$Zr_{0.526}Te_{1.00}$
$ZrTe_{2.0}$	$Zr_{0.700}Te_{1.40}$	$Zr_{0.600}Te_{1.20}$	$Zr_{0.500}Te_{1.00}$

vacancy concentration is summarized in Table XI, for the composition range ZrTe - $ZrTe_2$. The values tabulated, in Table XI, for 0% vacancies in the Te lattice are the same as those tabulated in Table VIII.

The Zr disorder parameter, which expresses the distribution of Zr vacancies on the Zr lattice sites (see Table IX), must be applied to these larger values of Zr vacancy concentration. For this series of calculations, the Zr disorder parameter was varied from 0.0 to 0.3. No attempt was made to apply a similar disorder parameter to the Te lattice. The Te vacancies were assumed to be distributed randomly on the Te lattice sites. The Te vacancy concentration ranged from 0 to 50%.

Theoretical relative intensities were calculated, using the (011) line as the standard. The results for the (012) line are shown in Figs. 12 to 15, for the (110) line in Figs. 16 to 19, and those for the (001) line are in Figs. 20 to 23. The percentages of Te vacancies are indicated on the curves; the value of the Zr disorder parameter is indicated on each figure. The corresponding experimental relative intensities are plotted only on the figures with a 0.0 value for the Zr disorder parameter.

For a given composition, introducing vacancies onto the Te lattice causes a decrease in the theoretical relative intensities of the (012) and (110) lines. Increasing the Zr disorder parameter causes an increase in the theoretical relative intensities for a given Te vacancy concentration.

TABLE XI

Mole Fraction of Zr Vacancies for Different Concentrations of Te Vacancies

Composition	Zr Vacancies (Mole Fraction)					
	Percentages of Vacant Te Sites					
	0%	10%	20%	30%	40%	50%
$ZrTe_{1.0}$	0.000	0.200	0.400	0.600	0.800	1.000
$ZrTe_{1.1}$	0.180	0.364	0.546	0.728	0.909	1.091
$ZrTe_{1.2}$	0.330	0.500	0.667	0.833	1.000	1.166
$ZrTe_{1.3}$	0.460	0.614	0.769	0.922	1.076	1.231
$ZrTe_{1.4}$	0.570	0.714	0.858	1.000	1.143	1.286
$ZrTe_{1.5}$	0.670	0.800	0.932	1.067	1.200	1.334
$ZrTe_{1.6}$	0.750	0.875	1.000	1.126	1.251	1.376
$ZrTe_{1.7}$	0.820	0.941	1.059	1.176	1.294	1.412
$ZrTe_{1.8}$	0.890	1.000	1.111	1.222	1.334	1.445
$ZrTe_{1.9}$	0.950	1.053	1.158	1.264	1.369	1.474
$ZrTe_{2.0}$	1.000	1.200	1.400	1.300	1.400	1.500

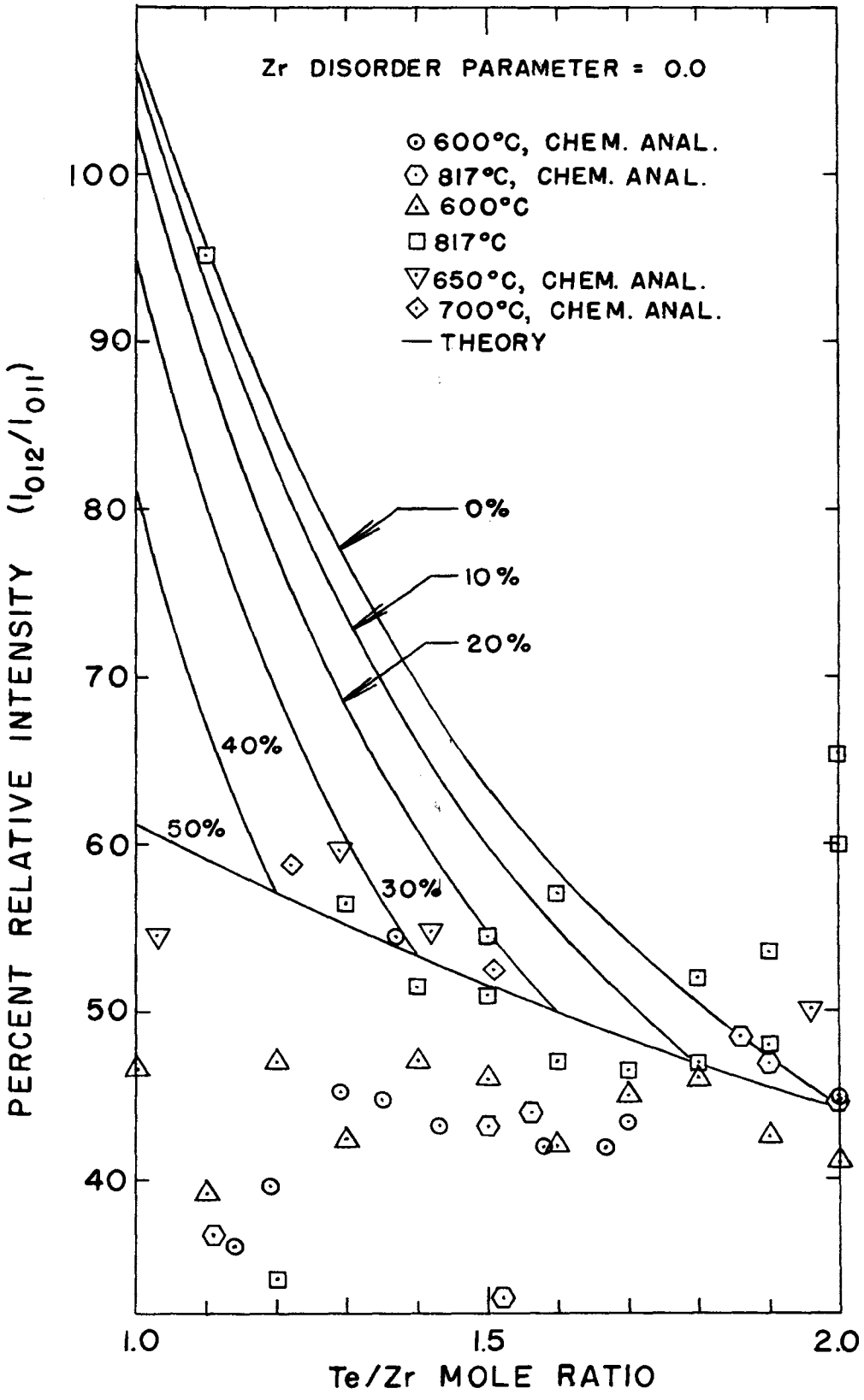

FIGURE 12 Theoretical and experimental values of I_{012}/I_{011}; Te vacancies with Zr disorder of 0.0.

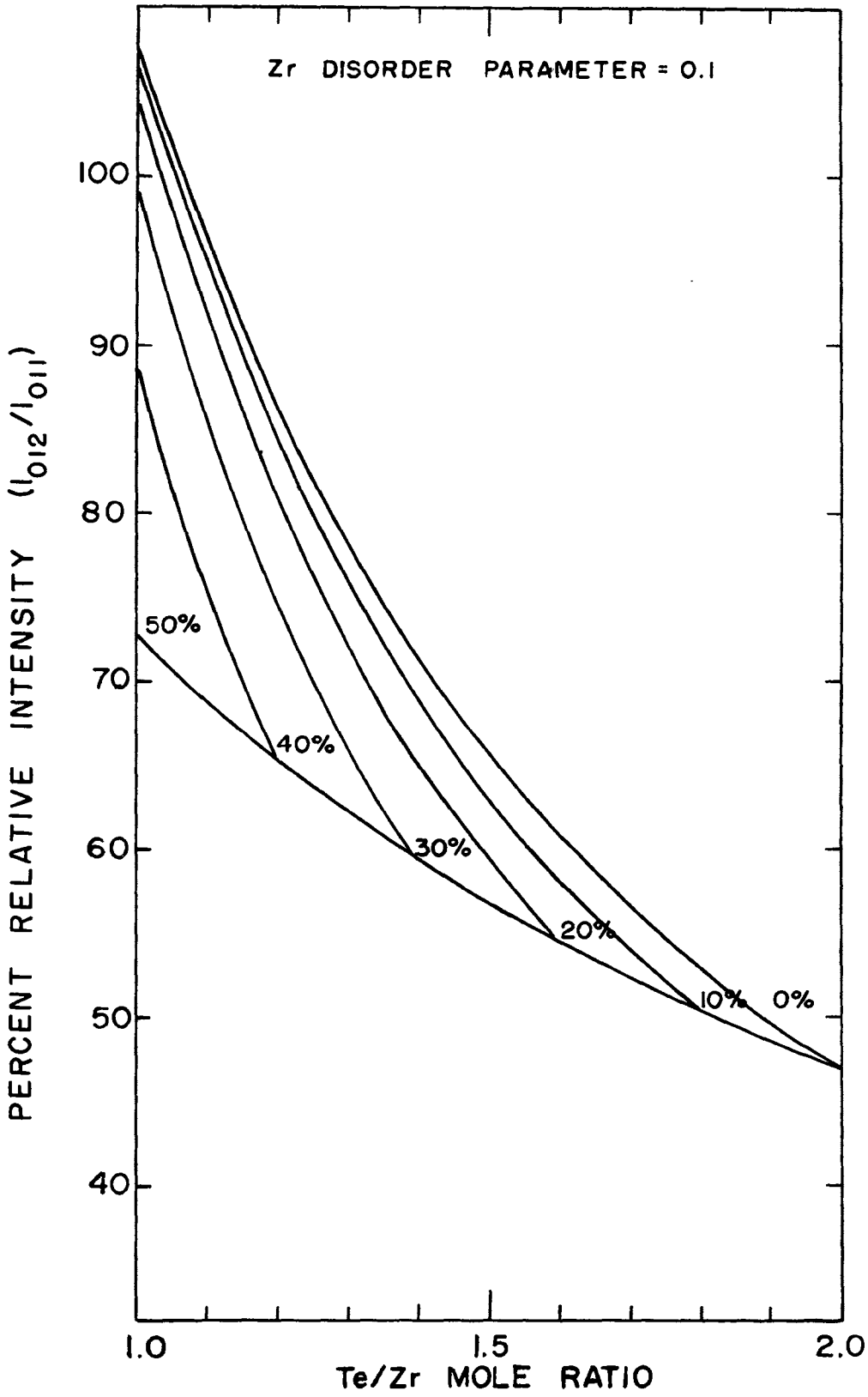

FIGURE 13 Theoretical values of I_{012}/I_{011}; Te vacancies with Zr disorder of 0.1.

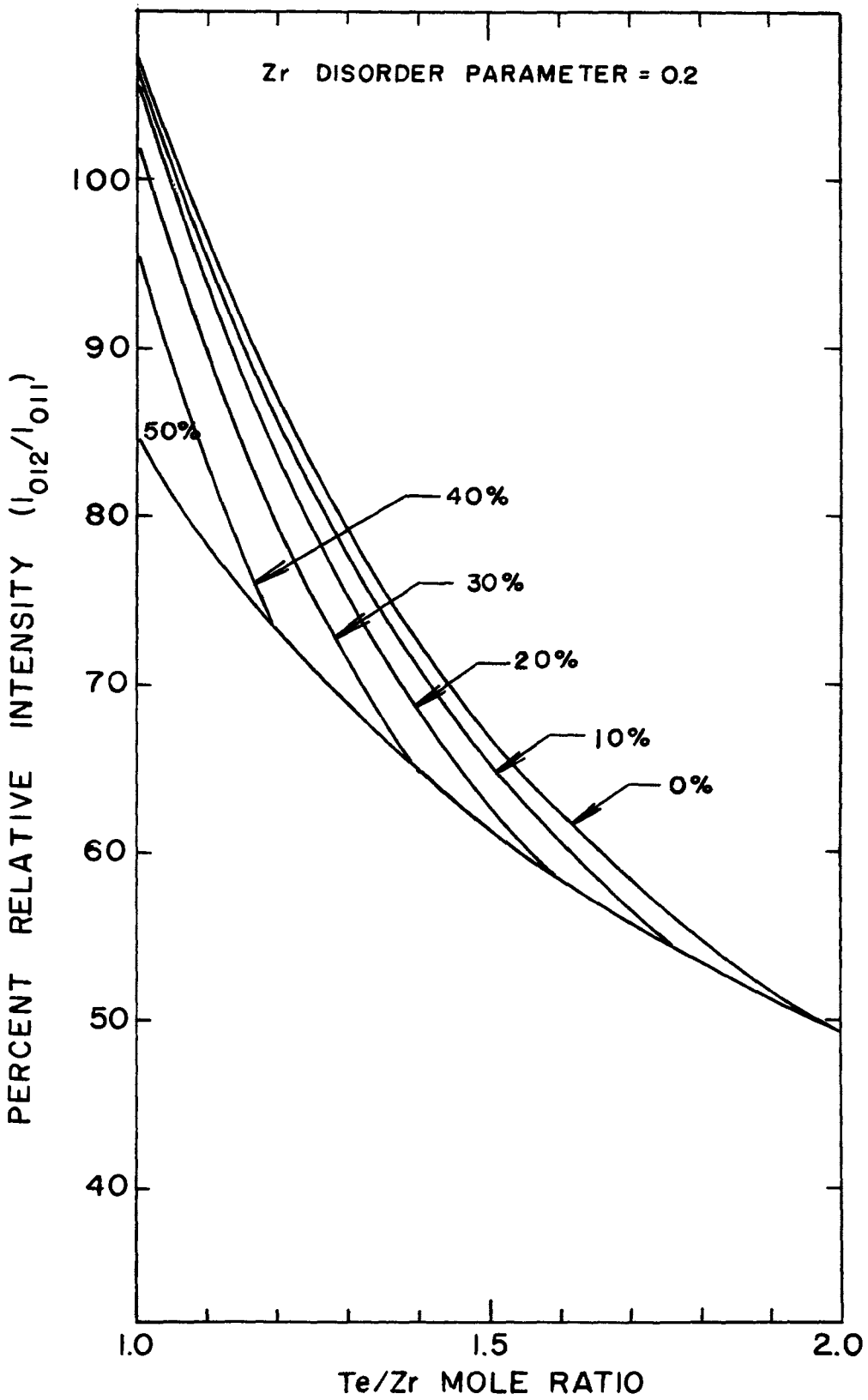

FIGURE 14 Theoretical values of I_{012}/I_{011}; Te vacancies with Zr disorder of 0.2.

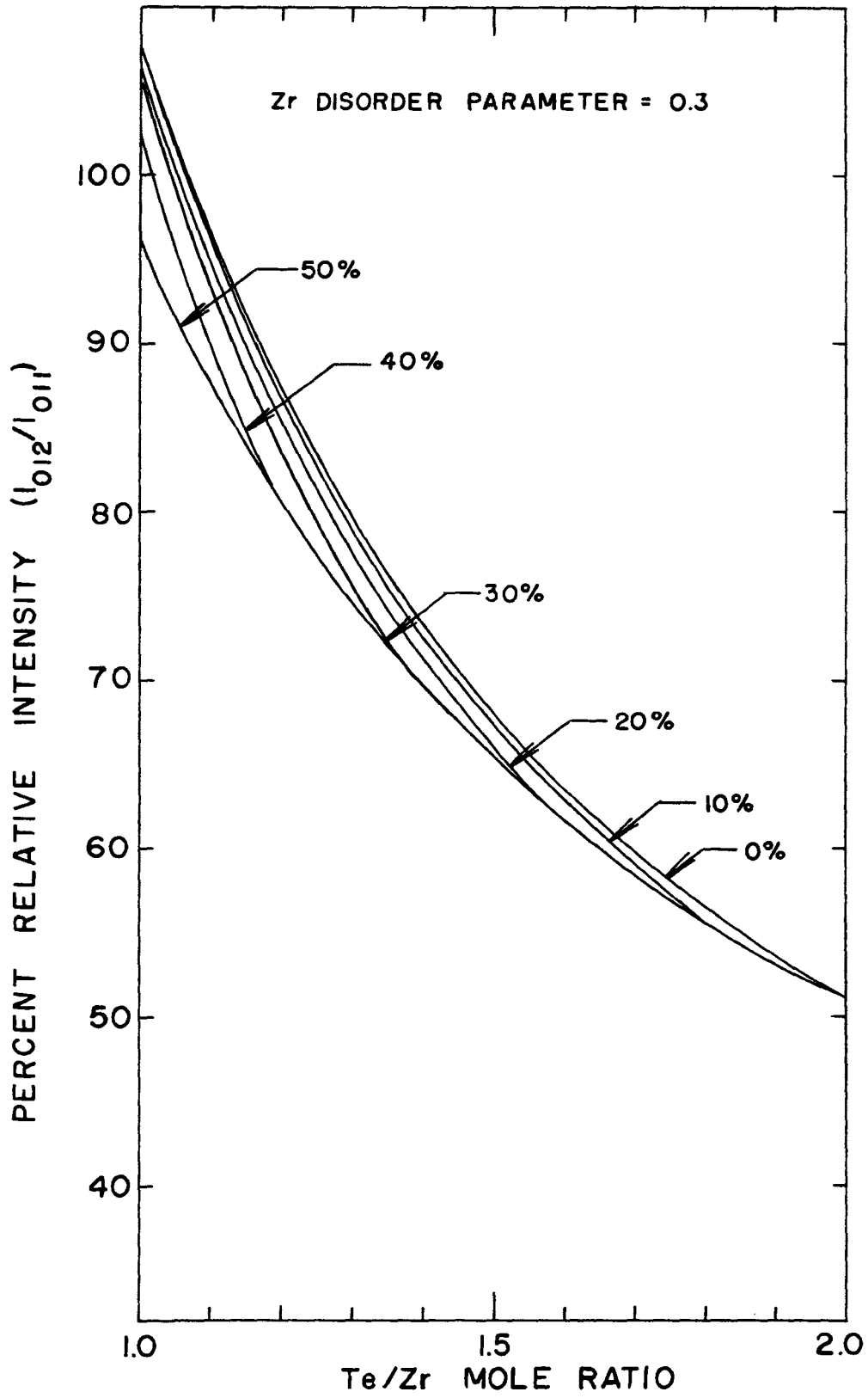

FIGURE 15 Theoretical values of I_{012}/I_{011}; Te vacancies with Zr disorder of 0.3.

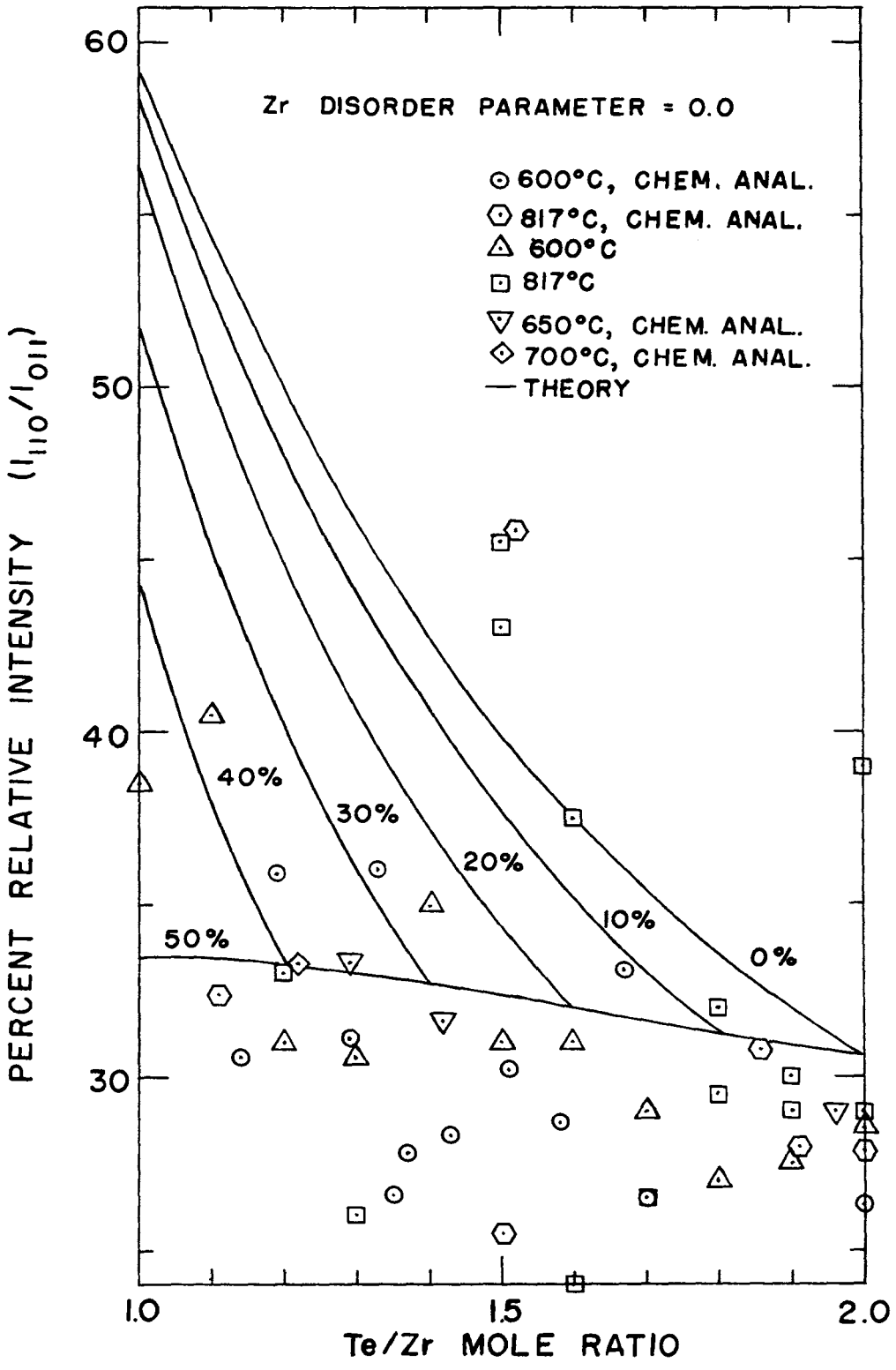

FIGURE 16 Theoretical and experimental values of I_{110}/I_{011}; Te vacancies with Zr disorder of 0.0.

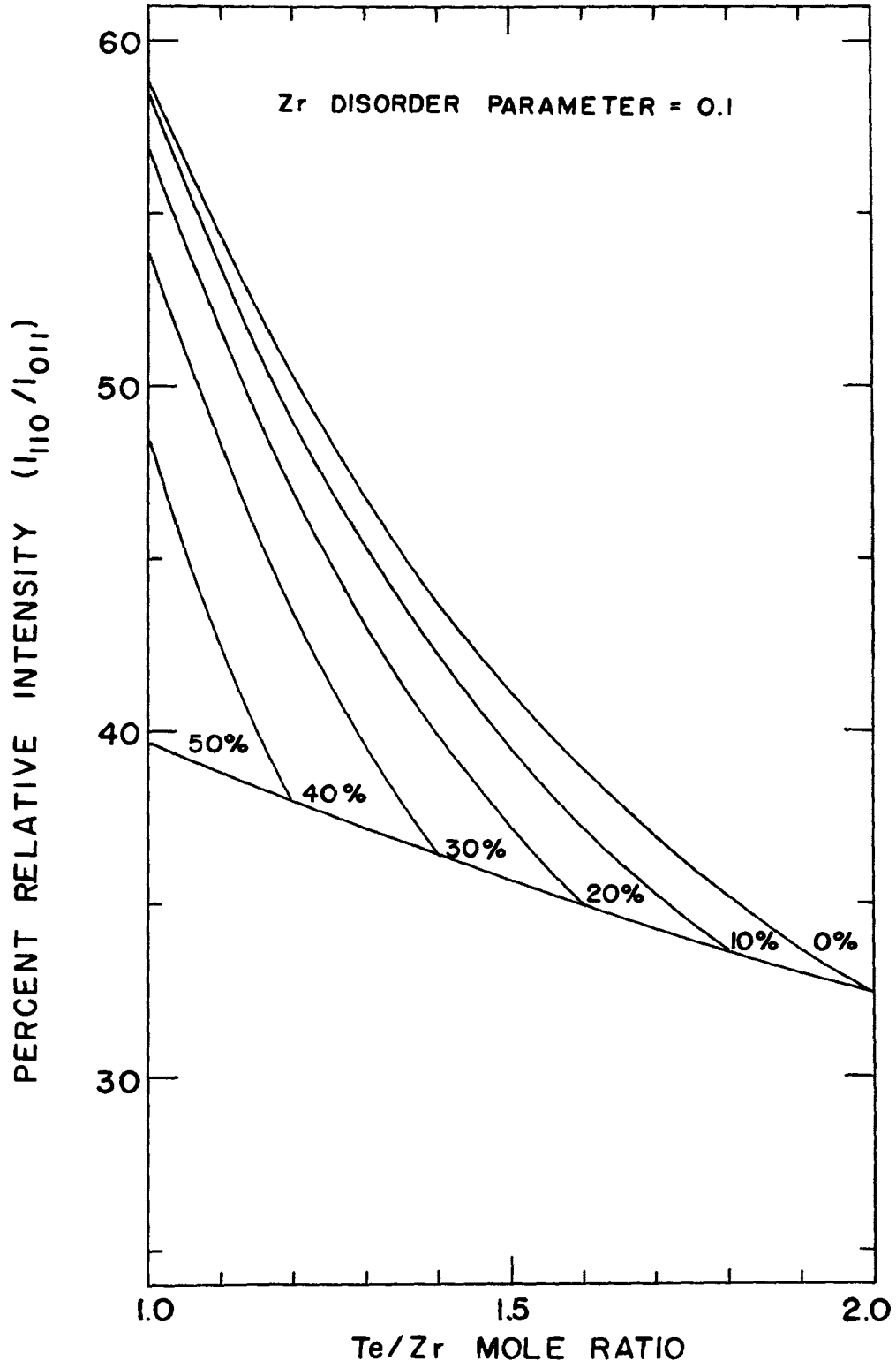

FIGURE 17 Theoretical values of I_{110}/I_{011}; Te vacancies with Zr disorder of 0.1.

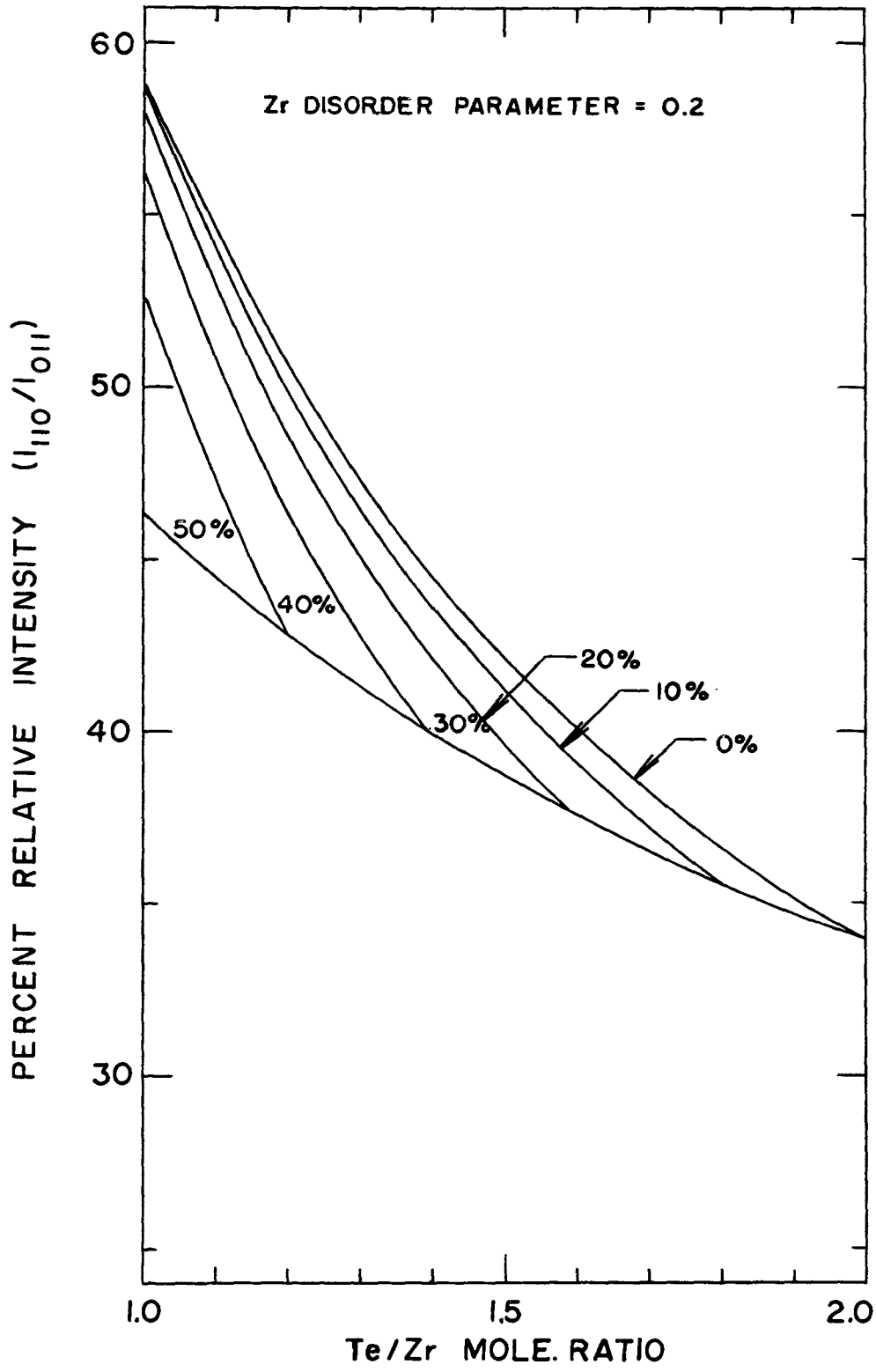

FIGURE 18 Theoretical values of I_{110}/I_{011}; Te vacancies with Zr disorder of 0.2.

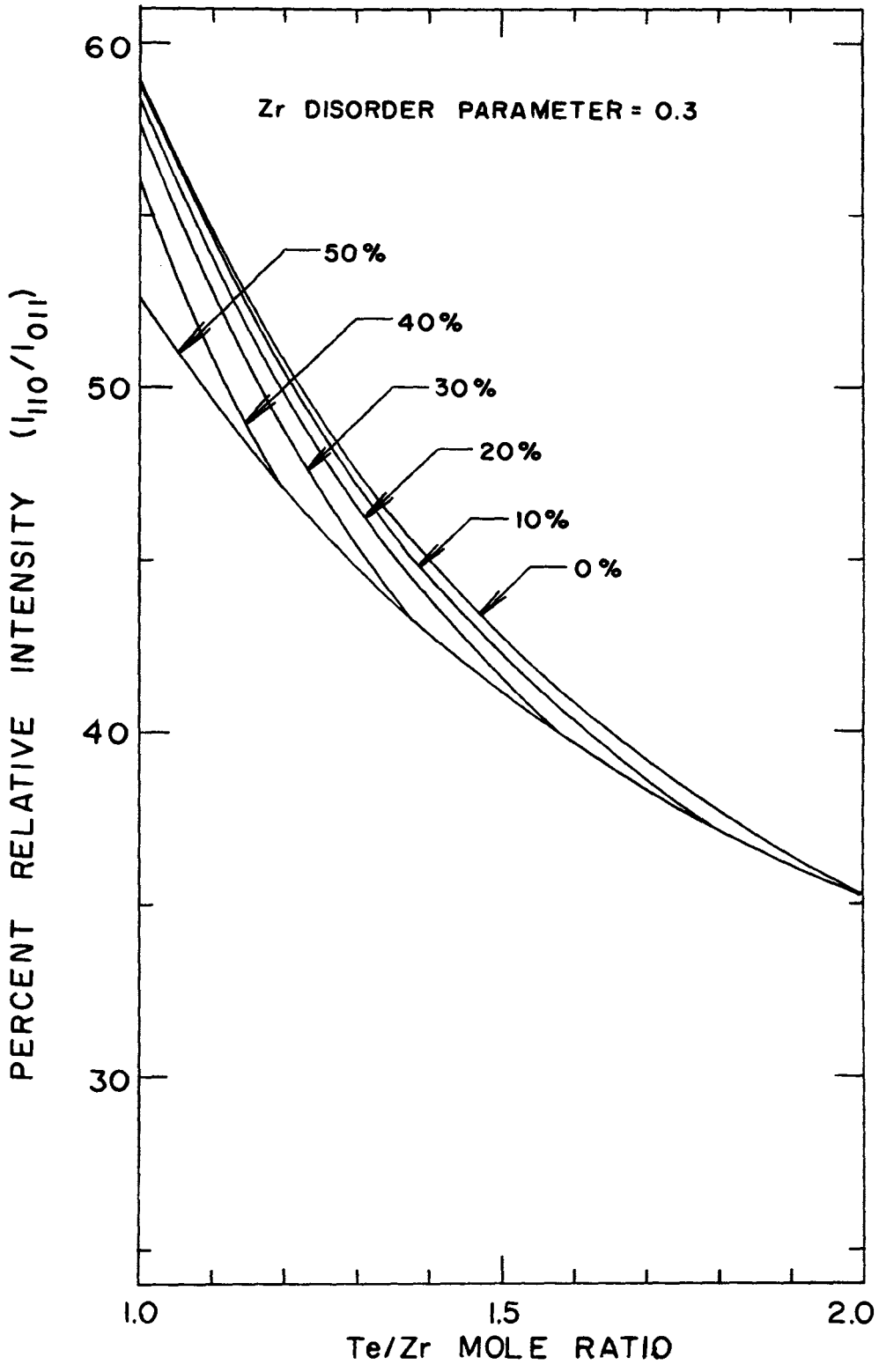

FIGURE 19 Theoretical values of I_{110}/I_{011}; Te vacancies with Zr disorder of 0.3.

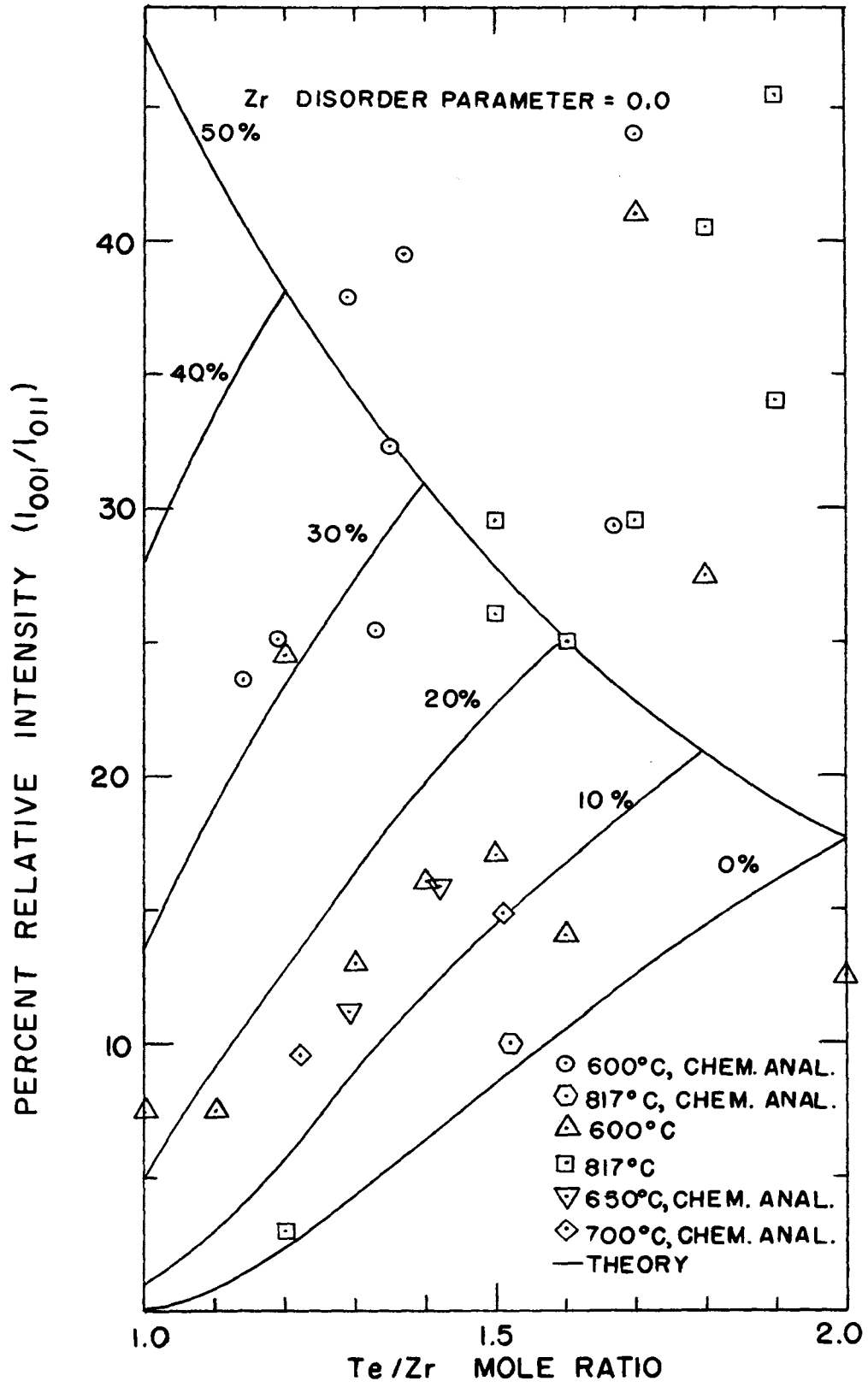

FIGURE 20 Theoretical and experimental values of I_{001}/I_{011}; Te vacancies with Zr disorder of 0.0.

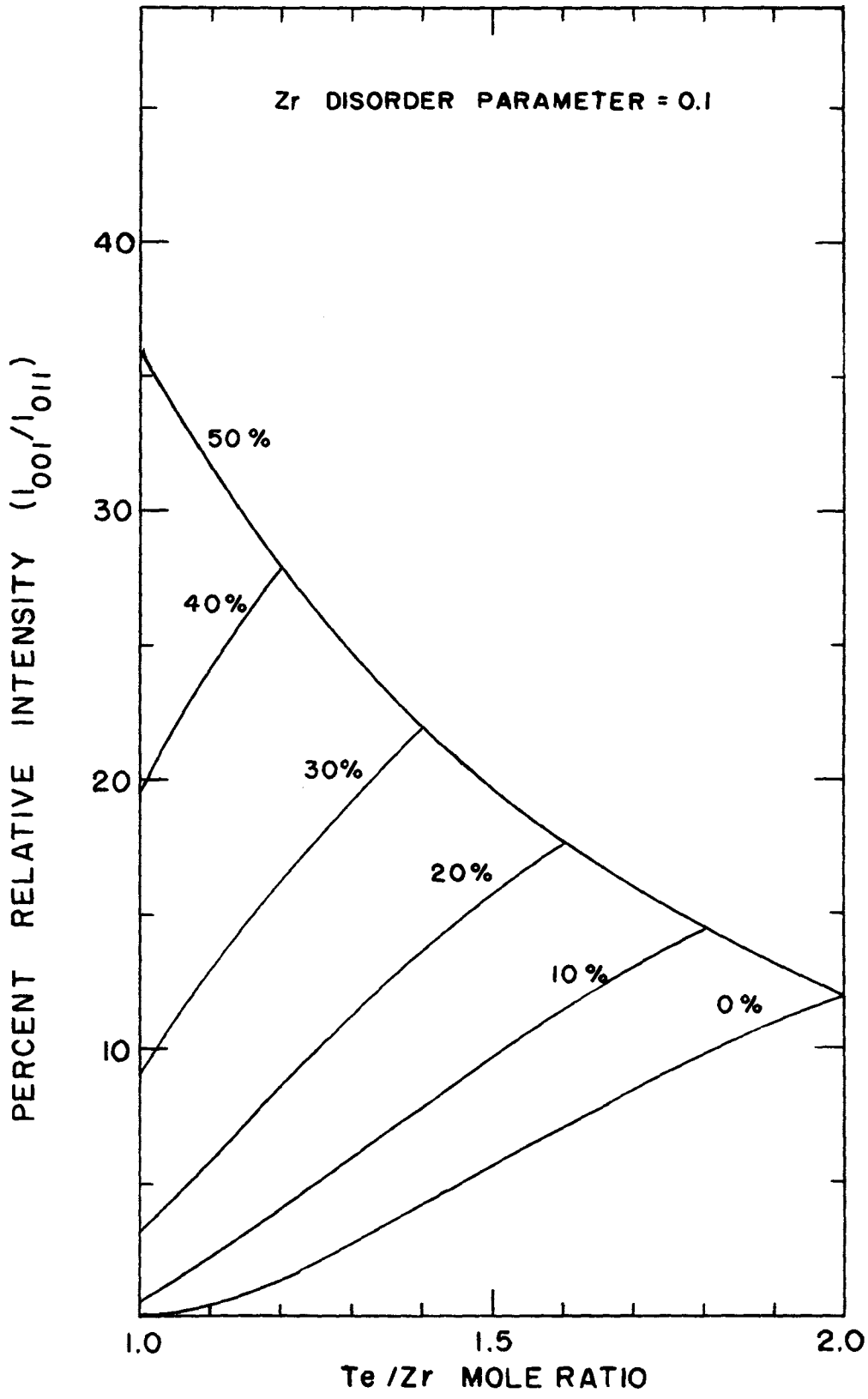

FIGURE 21 Theoretical values of I_{001}/I_{011}; Te vacancies with Zr disorder of 0.1.

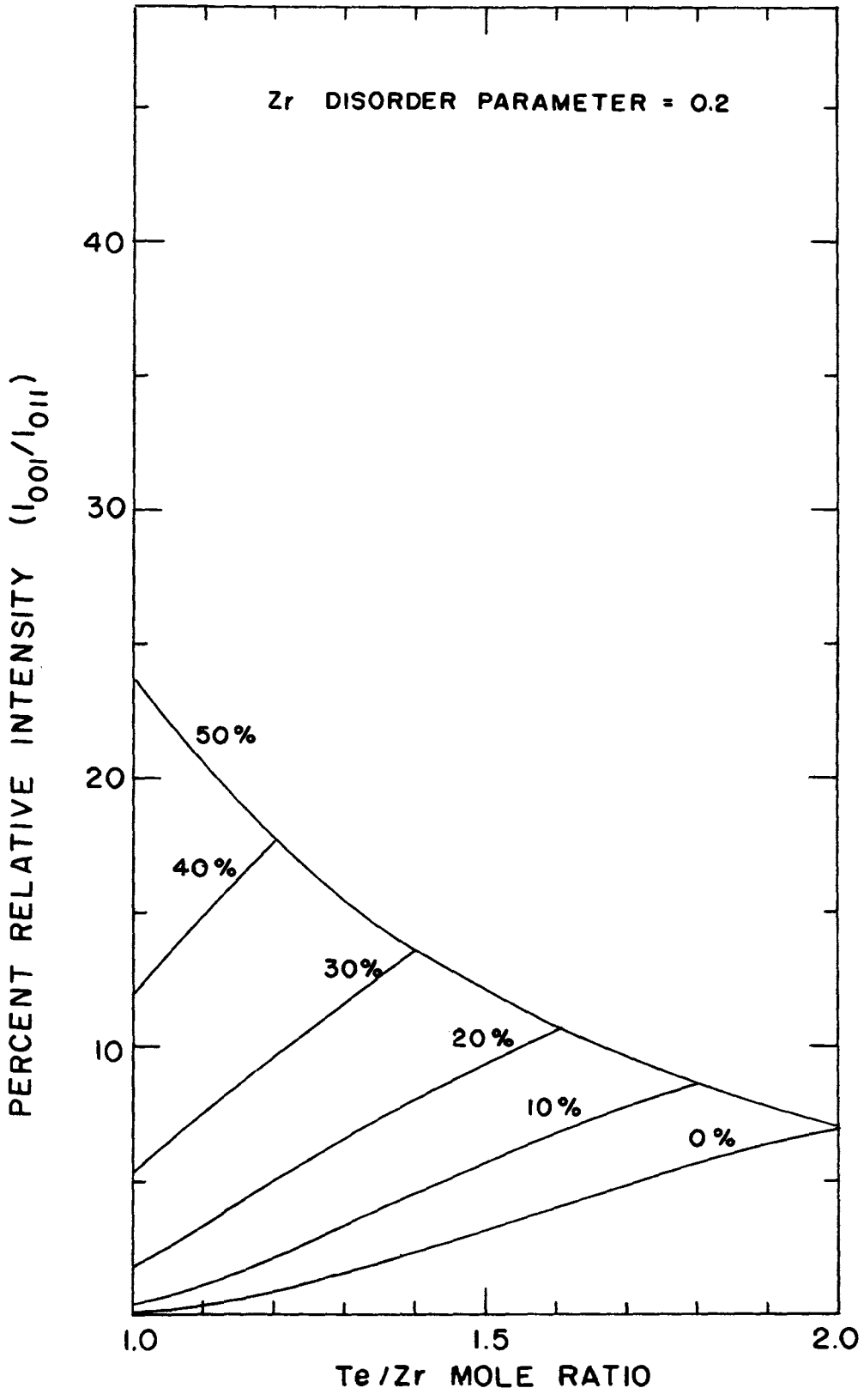

FIGURE 22 Theoretical values of I_{001}/I_{011}; Te vacancies with Zr disorder of 0.2.

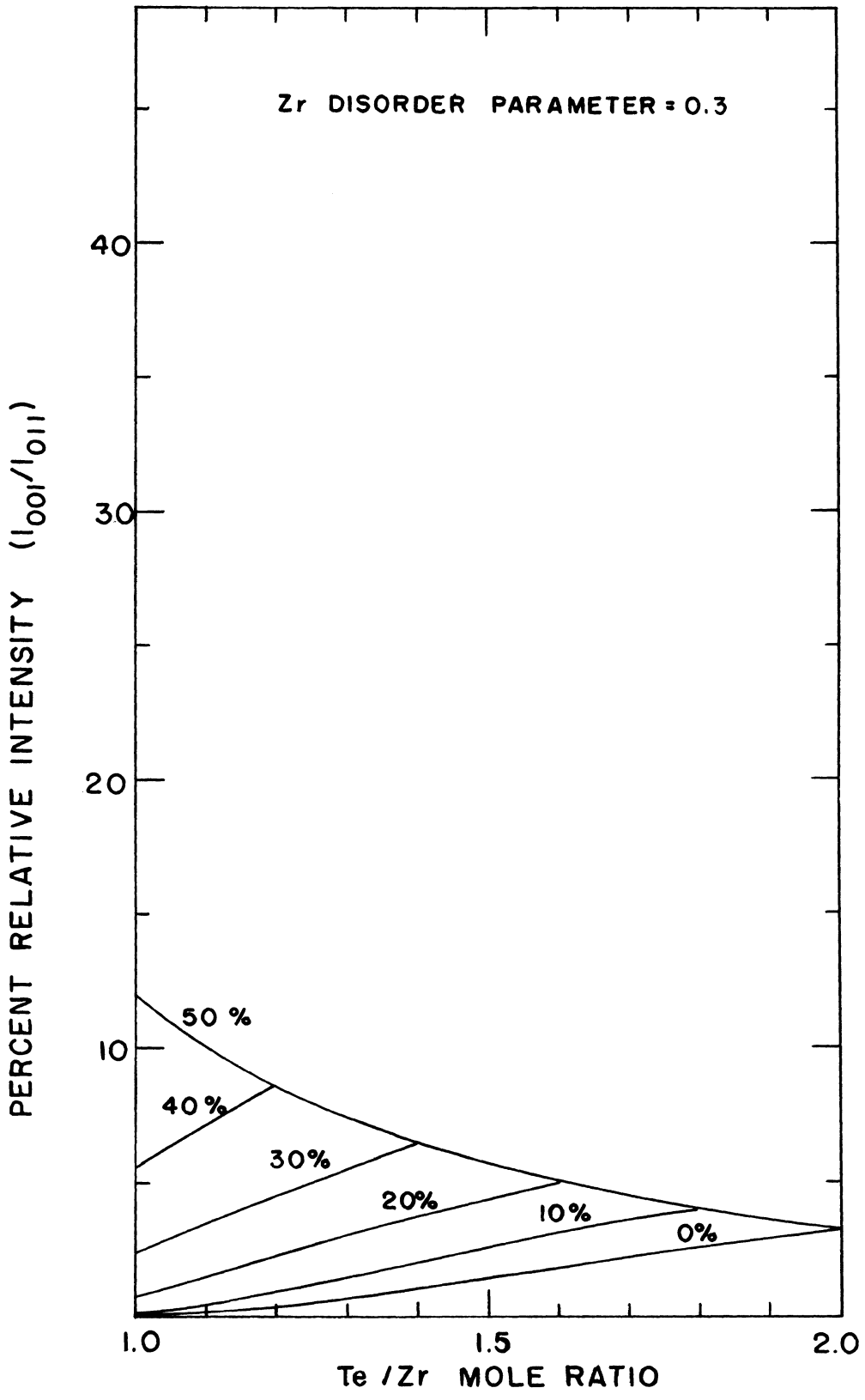

FIGURE 23 Theoretical values of I_{001}/I_{011}; Te vacancies with Zr disorder of 0.3.

Furthermore, the relative intensity curves coalesce. The point at which coalescence occurs is that point at which, for a given Te vacancy concentration, the "equivalent composition" as written equals that given by the Te/Zr mole ratio as written. From Table XI, we see that for 20% Te vacancies the "equivalent composition" as written equals that given by the Te/Zr mole ratio, as written, at the composition $ZrTe_{1.6}$. On Figs. 12 and 16, as well as all other figures in this series, the curve for 20% Te vacancies coalesces with the other relative intensity curves at $ZrTe_{1.60}$.

Introducing vacancies onto the Te lattice causes an increase in the theoretical relative intensities of the (001) line, as shown in Figs. 20 to 23. The relative intensity curves coalesce at the same points as in the previous figures. Increasing the Zr disorder parameter causes the theoretical relative intensities of the (001) line to decrease for a given Te vacancy concentration.

The experimental relative intensity results were plotted only on those figures having a Zr disorder parameter value of 0.0 because the discrepancy between the experimental and theoretical values increased with increasing values of the Zr disorder parameter. Most of the experimental values of I_{012}/I_{011} (Fig. 12) and I_{110}/I_{011} (Fig. 16) are below the theoretical values for a Zr disorder parameter value of 0.0. This indicates that preferred orientation, rather than an incorrect vacancy model, caused the lack of agreement between the experimental and theoretical values

shown in Figs. 8 and 9. Not all the experimental results for the (001) line could be plotted in Fig. 20, because many values were considerably higher than 49%. This again indicates that preferred orientation occurred in the samples.

As done twice previously, to remove the effect of preferred orientation in the sample, theoretical values of I_{001}/I_{002} were calculated for several values of the Te vacancy concentration and of the Zr disorder parameter. The results of these calculations, with the corresponding experimental values, are shown in Figs. 24 to 27.

The figures show that the assumption of Te vacancies, in addition to the assumption of degrees of disorder in the Zr vacancy distribution, brings all experimental relative intensities within the region bounded by the theoretical values. In Fig. 23, an assumption of 10% Te vacancies with a Zr disorder parameter of 0.0 is sufficient to encompass all experimental points outside the region of theoretically calculated values in Fig. 11. However, because the vast majority of experimental points in Fig. 11 indicated partial disordering of the Zr vacancies, it is reasonable to assume that there is also partial disordering of Zr vacancies in the compositions represented by points outside the region encompassed by the theoretical values.

Consider the data for chemically analyzed samples only and assume that there is disordering of the Zr vacancies. Most of the data in Fig. 25 agree with a structural model of 10% Zr vacancies at z = 0 (Zr disorder parameter = 0.1)

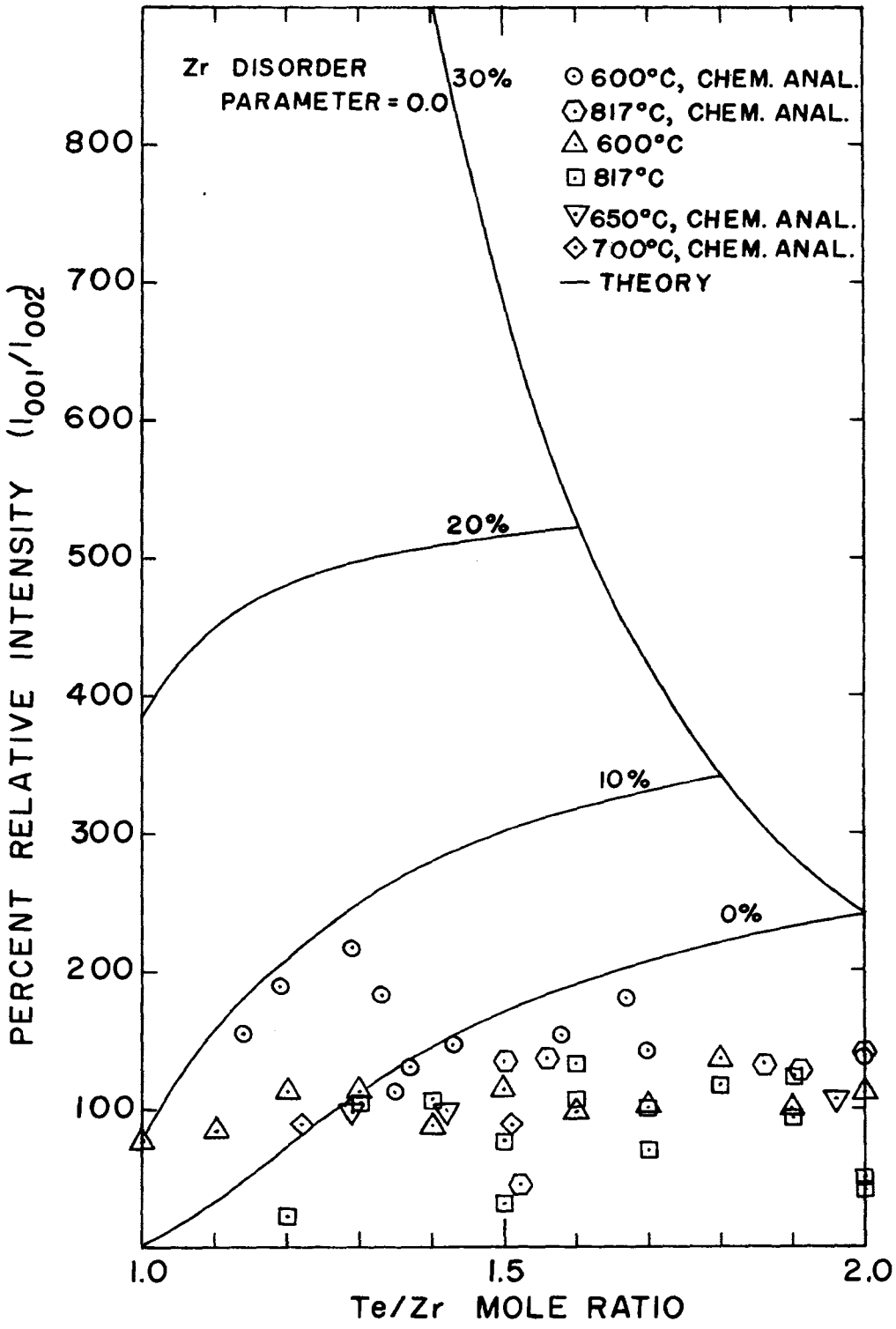

FIGURE 24 Theoretical and experimental values of I_{001}/I_{002}; Te vacancies with Zr disorder of 0.0.

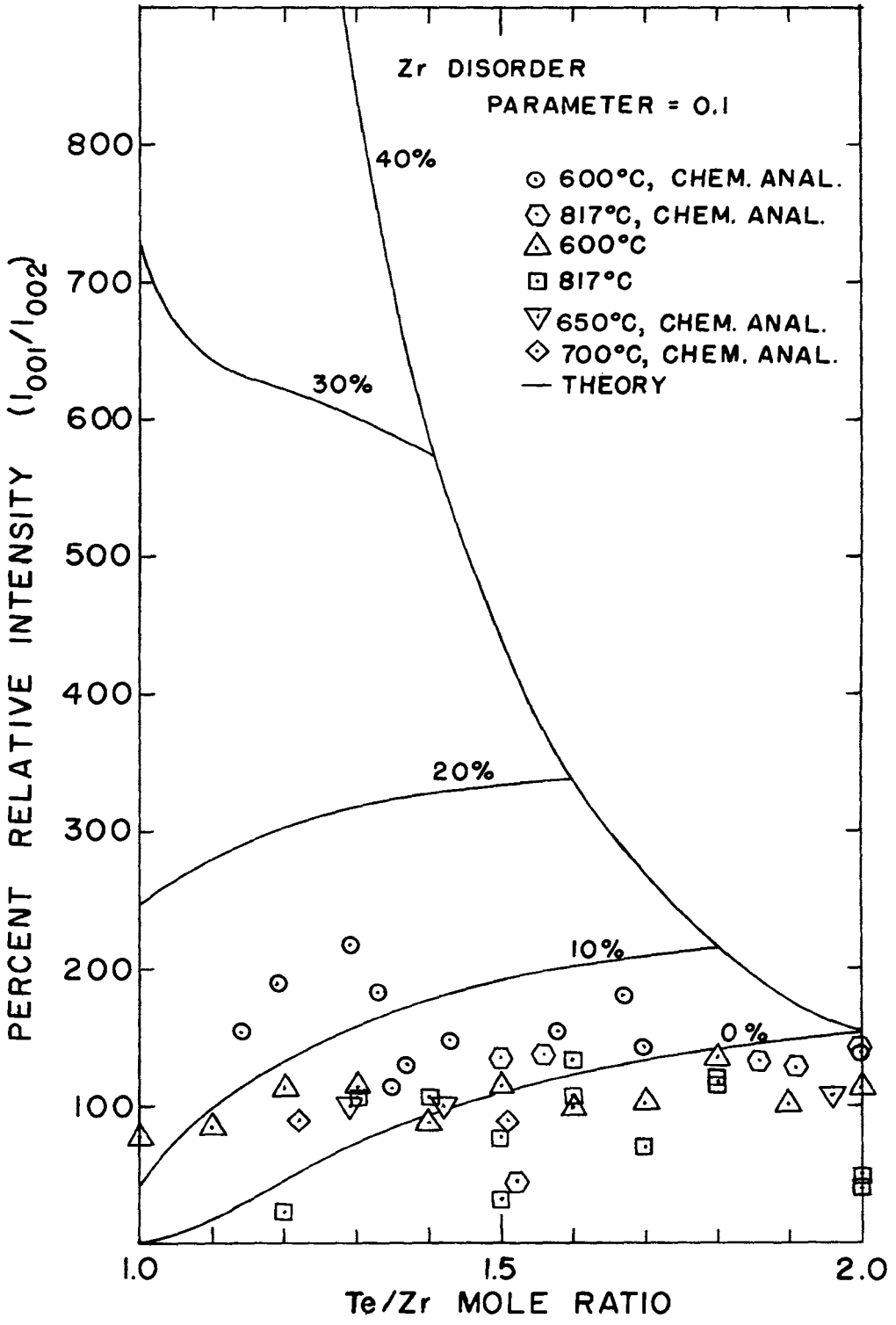

FIGURE 25 Theoretical and experimental values of I_{001}/I_{002}; Te vacancies with Zr disorder of 0.1.

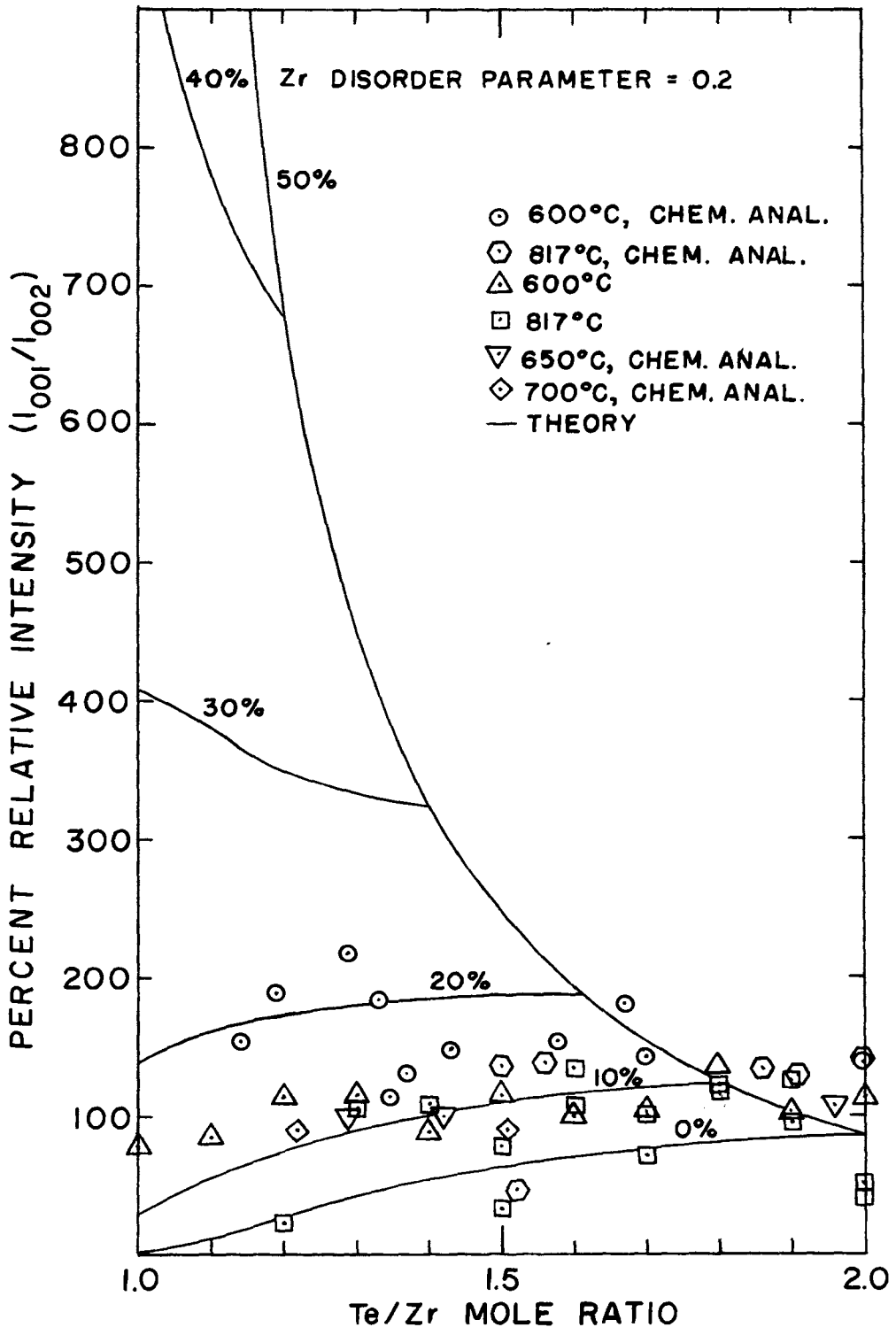

FIGURE 26 Theoretical and experimental values of I_{001}/I_{002}; Te vacancies with Zr disorder of 0.2.

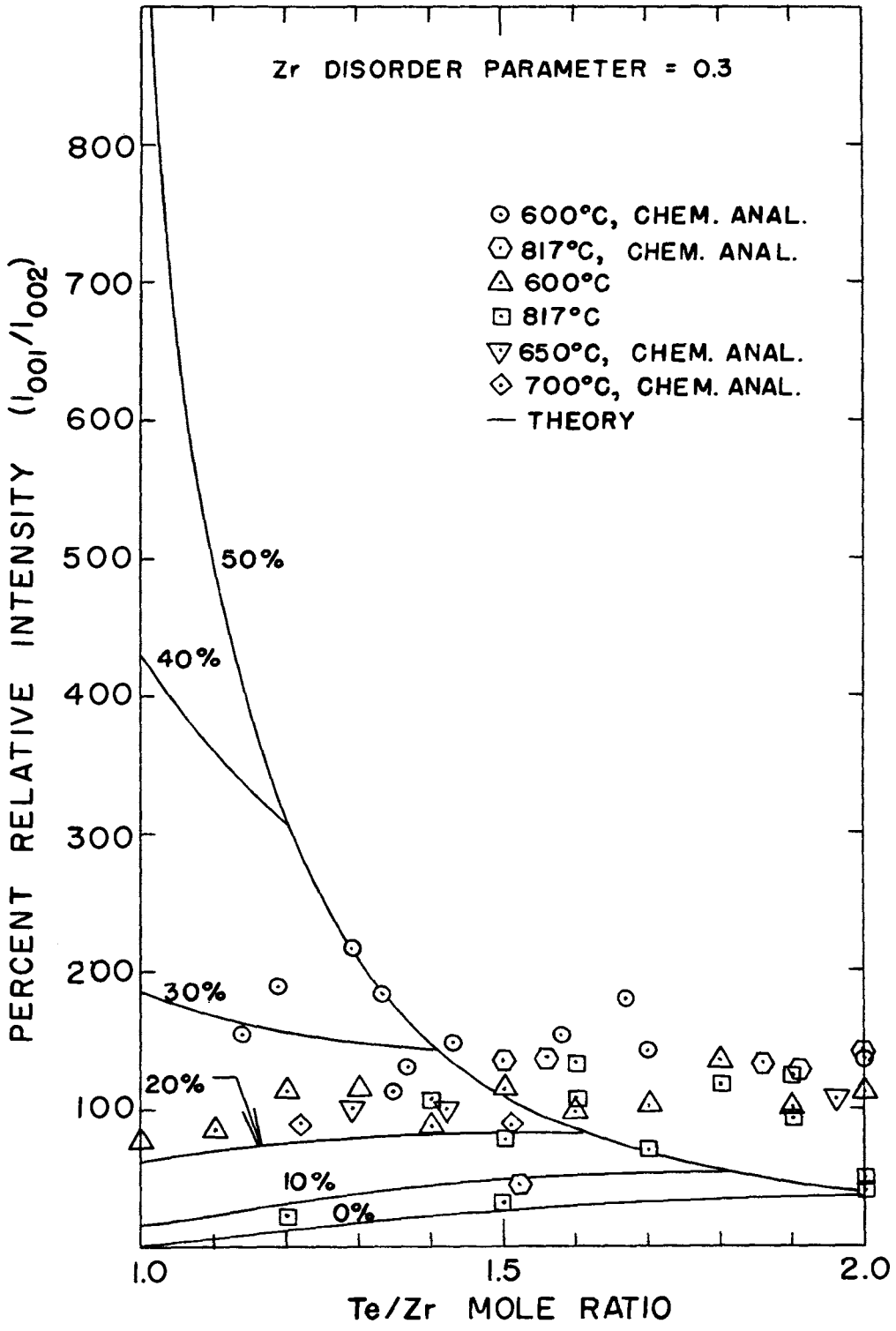

FIGURE 27 Theoretical and experimental values of I_{001}/I_{002}; Te vacancies with Zr disorder of 0.3.

and up to 15% Te vacancies. All of the 600°C data fit this model except $ZrTe_2$. Samples in the composition range $ZrTe_{1.5}$ - $ZrTe_2$ fired at temperatures higher than 600°C do not fit this model, however.

Most of the data also agree with a structural model with 20% Zr vacancies at z = 0 (Fig. 26, Zr disorder parameter = 0.2) and up to 23% Te vacancies. Figs. 25 and 26 show that the data for the composition range $ZrTe_{1.5}$ - $ZrTe_2$ would agree quite well with a structural model of 15% Zr vacancies at z = 0 (Zr disorder parameter = 0.15) and 0 to 5% Te vacancies. Only one experimental point would be in disagreement with this model.

Data for the composition range ZrTe - $ZrTe_{1.5}$ agree with an additional structural model, with 30% Zr vacancies at z = 0 (Fig. 27, Zr disorder parameter = 0.3) and up to 35% Te vacancies.

It is much easier to assign a specific structural model to compositions in the region $ZrTe_{1.5}$ - $ZrTe_2$ because there is much less overlapping of the theoretical relative intensities for different assumed values of Zr disorder and Te vacancy concentration in this composition region. In order to assign specific defect structures to compositions in the range ZrTe - $ZrTe_{1.5}$, auxiliary experiments must be made. Density measurements seem to hold the most promise for success in evaluating these defect structures. Because of the reactivity of the samples with water and other liquids, a gas pycnometer, utilizing a dry inert gas, should be used

to make density measurements.

These results agree with the findings of McTaggart and Wadsley[5] and of Hahn and Ness.[4] McTaggart and Wadsley had to assume a defective anionic lattice, specifically 5% Te vacancies (Tables I and X), to obtain agreement between experimental and theoretical density values for the $ZrTe_{1.7}$ Sample. Hahn and Ness[4] found that pycnometric densities were considerably lower than X-ray densities, consistent with a model of Te vacancies.

D. Lattice Parameter Measurements

The (200) line of MgO was used as the internal standard. The d-spacing[11] of this line is between the d-spacings of the (003) and (110) lines of the telluride compositions and within 7° 2θ of the (013) line of the telluride compositions, used to obtain the c_o parameter when the (003) line was weak. Furthermore, all lines are in a 2θ region where an error in angle of 0.01° 2θ results in an error of only 0.0005 Å. in the d-spacing. Scatter in the data was greater than 0.0005 Å.

The results of lattice parameter measurements on samples fired at 600°C are presented in Figs. 28 and 29, plotted as a function of analyzed composition and intended composition, respectively. A limited amount of data was obtained on samples fired at 817°C; this is presented in Figs. 30 and 31, also plotted as a function of analyzed and intended composition, respectively. The data of Hahn and

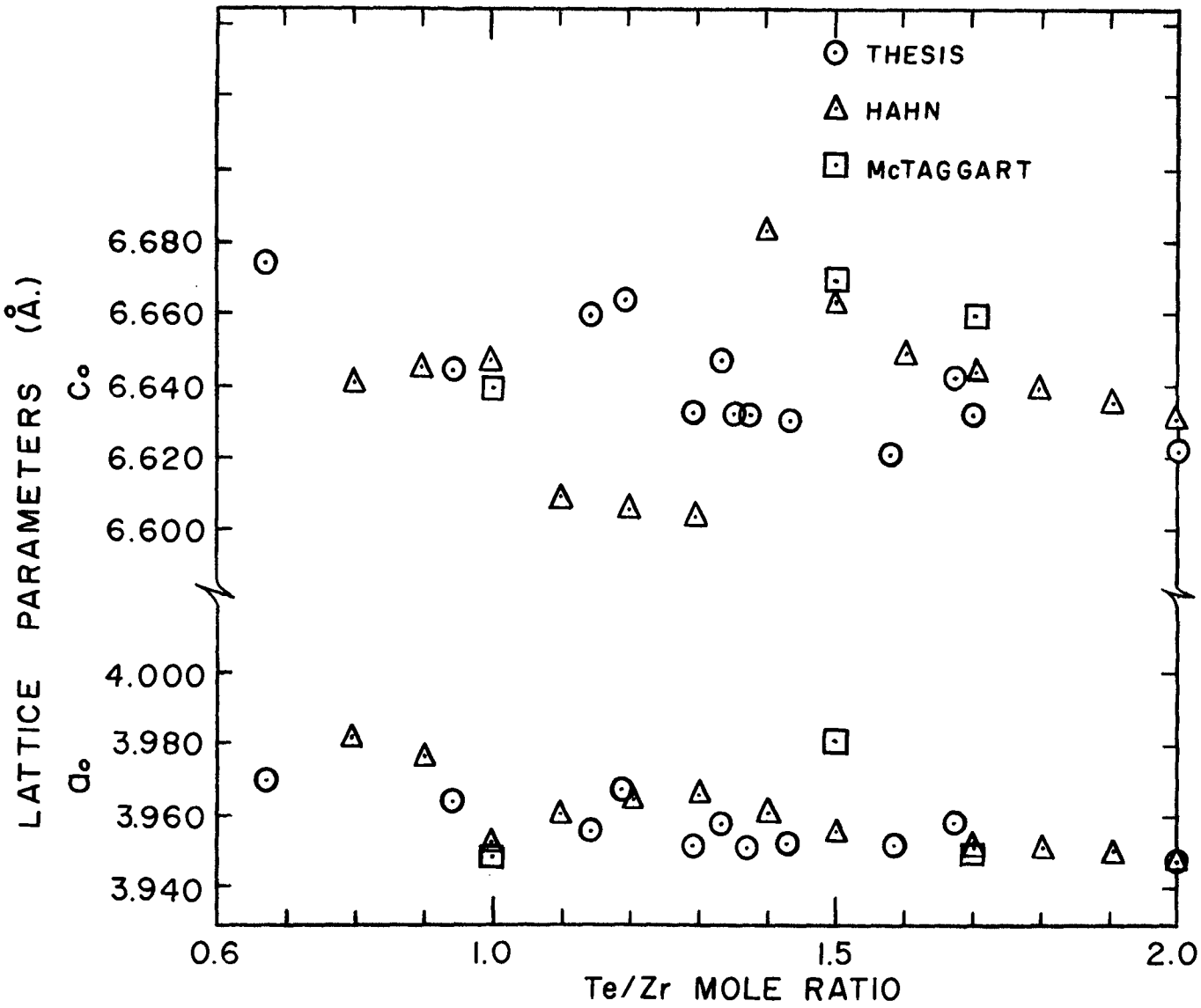

FIGURE 28 The effect of Te content on the lattice parameters, 600°C firing. Composition determined by chemical analysis.

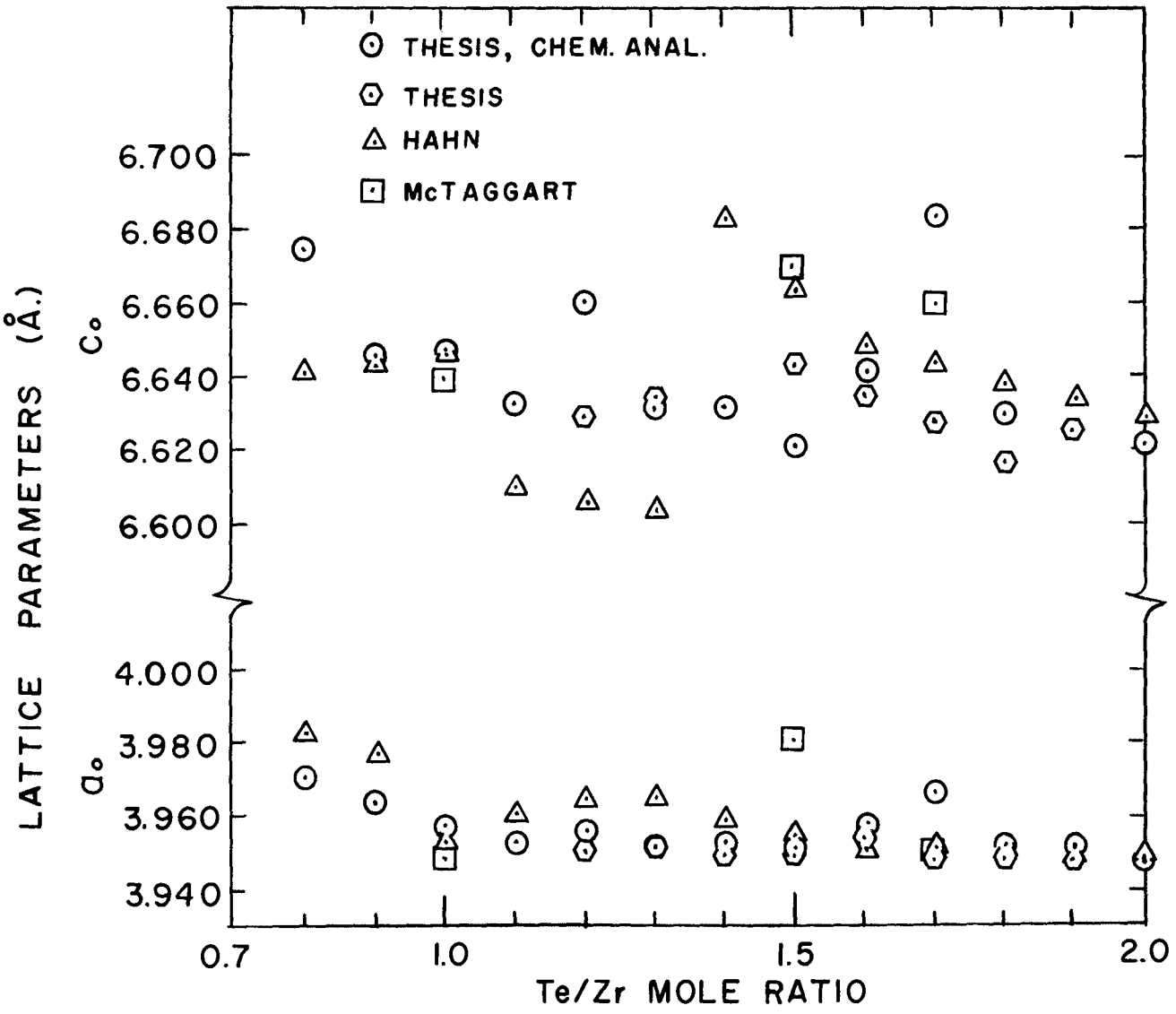

FIGURE 29 The effect of Te content on the lattice parameters, 600°C firing. Composition based on original formulation.

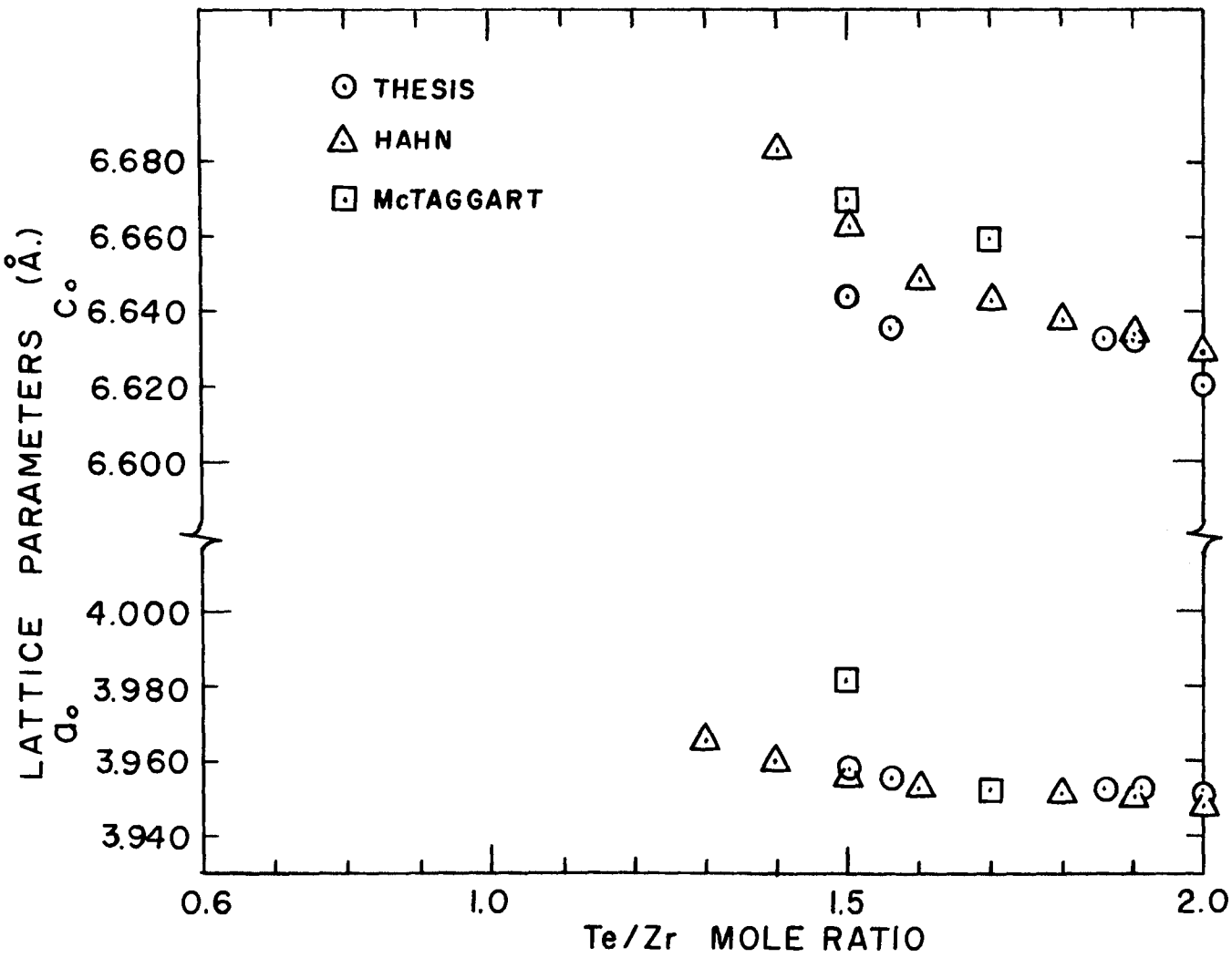

FIGURE 30 The effect of Te content on the lattice parameters, 817°C firing. Composition determined by chemical analysis.

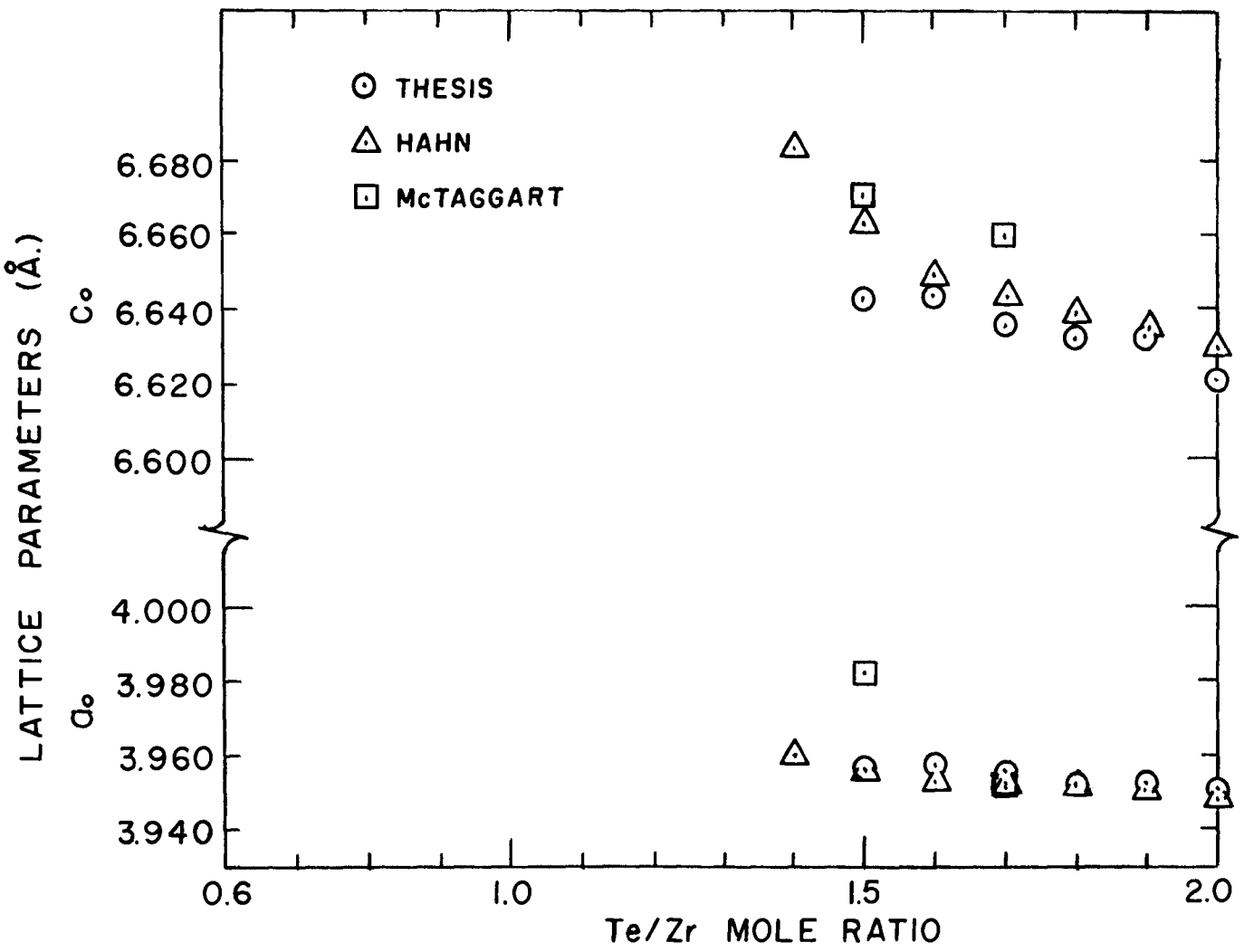

FIGURE 31 The effect of Te content on the lattice parameters, 817°C firing. Composition determined by original formulation.

Ness,[4] as well as the few points from the work of McTaggart and Wadsley,[5] are included.

One sample from the 600°C firing (1.5-1) and three samples from the 817°C firing (1.5-5, 1.6-6, and 1.7-4) were two-phase, i.e., the hard metallic material and the powder sinter. There was insufficient material to make separate X-ray scans, including lattice parameter measurements, on each phase, so the scans were made on the mixed phases. The value selected for the "analyzed composition" was an average of the chemical analysis results for the separate phases. Because the 1.5-5s chemical analysis specimen was dropped, no lattice parameter values were plotted in Fig. 30 for this sample. They have been plotted, however, in Fig. 31.

When the lattice parameter data for samples fired at 600°C are plotted as a function of analyzed composition (Fig. 28), there is good agreement between the a_o values of Hahn and Ness[4] and those of this work. Although there is some scatter, a_o can be seen to increase with decreasing Te content. The c_o values are in general agreement with those of Hahn and Ness,[4] although there is considerable scatter in the present data and those of Hahn and Ness.[4] When the data for samples fired at 600°C are plotted as a function of intended composition (Fig. 29), there is much more scatter in the present results, especially in the c_o values. In view of the difficulties encountered in the mixing of these samples, the chemically analyzed compositions are to

be preferred as a basis for plotting the results.

When the lattice parameter data for samples fired at 817°C are plotted as a function of analyzed composition (Fig. 30), there is good agreement between the a_o values of Hahn and Ness[4] and those of this work. The results for c_o are in good agreement with those of Hahn and Ness[4] in the $ZrTe_2$ region but not in the $ZrTe_{1.5}$ region. When the data for the samples fired at 817°C are plotted as a function of intended composition (Fig. 31), there is still good agreement between the a_o values of Hahn and Ness[4] and those of this work. The results for c_o are in fair agreement with those of Hahn and Ness[4] and in much better agreement than the corresponding results at 600°C. The large scatter in the lattice parameter measurements, especially the c_o values, is indicative of the difficulties of working in the Zr-Te system.

The data represented by the octagons in Fig. 29 were obtained on 600°C samples that had been stored in closed vials for almost one year. Many samples produced a popping sound when the vials were opened, as if pressure had built up inside the vial. There was an odor that was probably the hydrogen telluride odor reported by Hahn and Ness[4] and McTaggart and Wadsley.[5] In a few of the year-old samples, the (003) and (013) lines had completely disappeared, but the (110) line was still present. Meanwhile, three new lines had appeared in the 10° 2θ range normally scanned. Other samples in this year-old group showed much reduced

(003) and (013) lines as well as the three new lines. Full X-ray scans (0.2° 2θ/min.) were obtained for three of the year-old samples (2.0-3, 1.8-3, and 1.4-3), which exhibited no (003) and (013) lines. The results of these scans are summarized in Appendix K. The patterns obtained were consistent with that of Te; there were no Zr peaks in the diffraction pattern. This raises some interesting questions as to what chemical compound caused the odor and relates to the structural state of the Zr.

Year-old samples which had been fired at 817°C for 130 hours exhibited no X-ray lines. These materials were free-flowing in the sample vials and did not produce a popping sound when the vial was opened. It is uncertain as to whether these samples degraded differently from those fired at 600°C or the vials were not completely sealed.

E. Hydration Measurements

The results of the hydration studies are presented in Figs. 32 to 36. The data were plotted as "percent weight gained" rather than as "moles of water gained", because chemical analyses revealed that the composition of individual samples was considerably different from the value originally intended.

Both Hahn and Ness[4] and McTaggart and Wadsley[5] stated that the Zr-tellurides pick up oxygen; this study indicates that they pick up water. Sample 1.7-2 (Fig. 32) showed a sudden weight loss at 650 hours and again at about 950

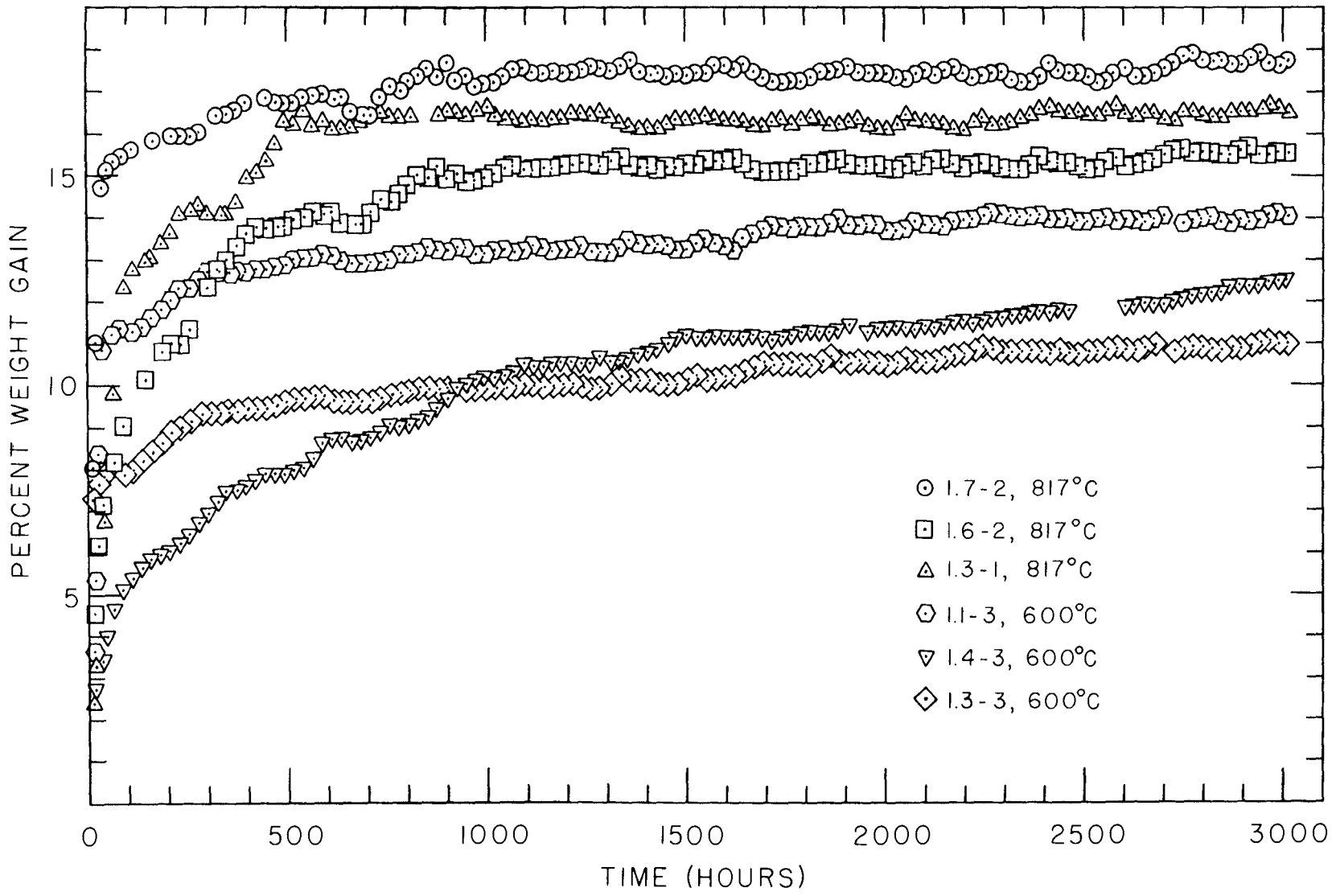

FIGURE 32 Weight gain data for samples 1.7-2, 1.6-2, 1.3-1, 1.1-3, 1.4-3, and 1.3-3.

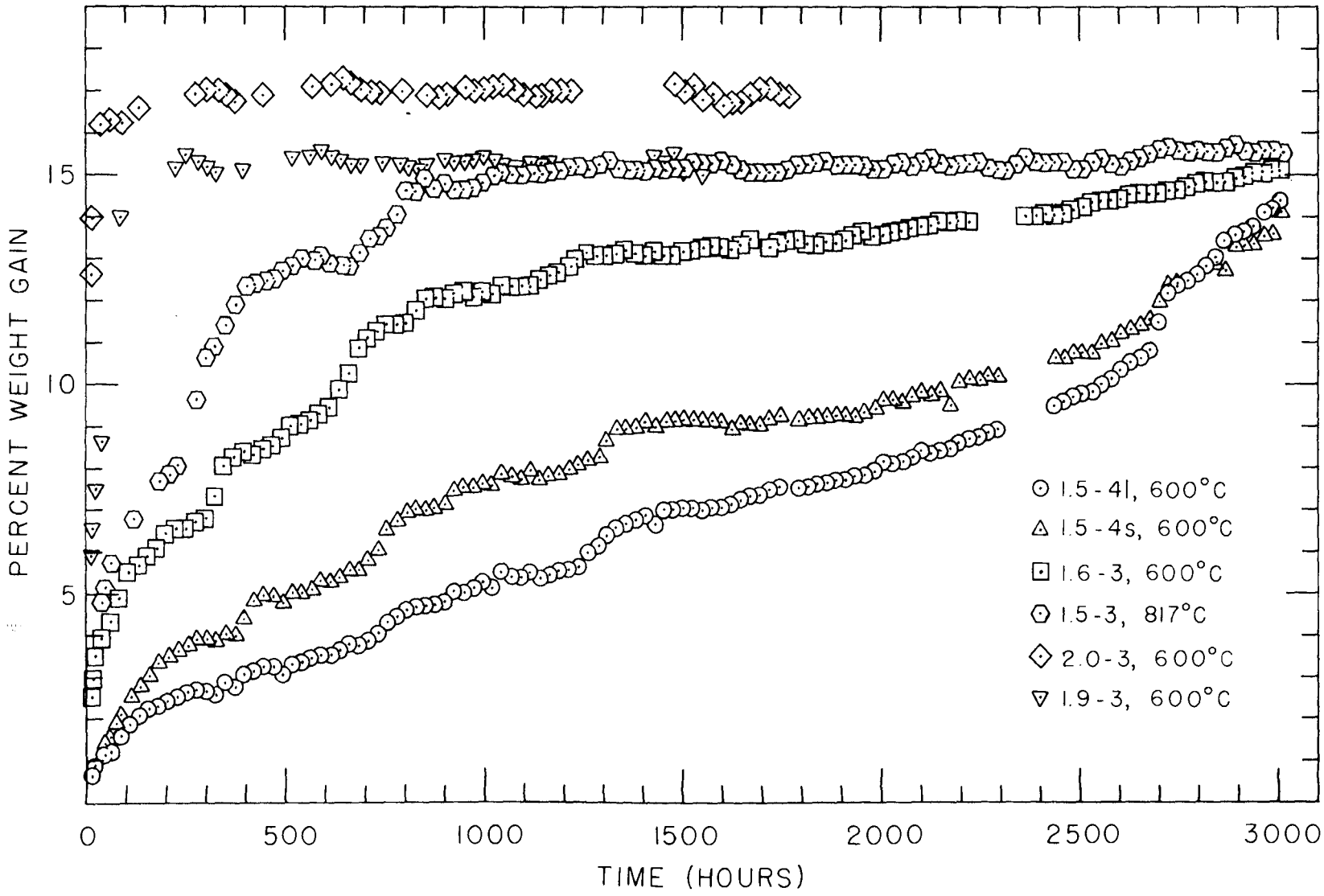

FIGURE 33 Weight gain data for samples 1.5-4ℓ, 1.5-4s, 1.6-3, 1.5-3, 2.0-3, and 1.9-3.

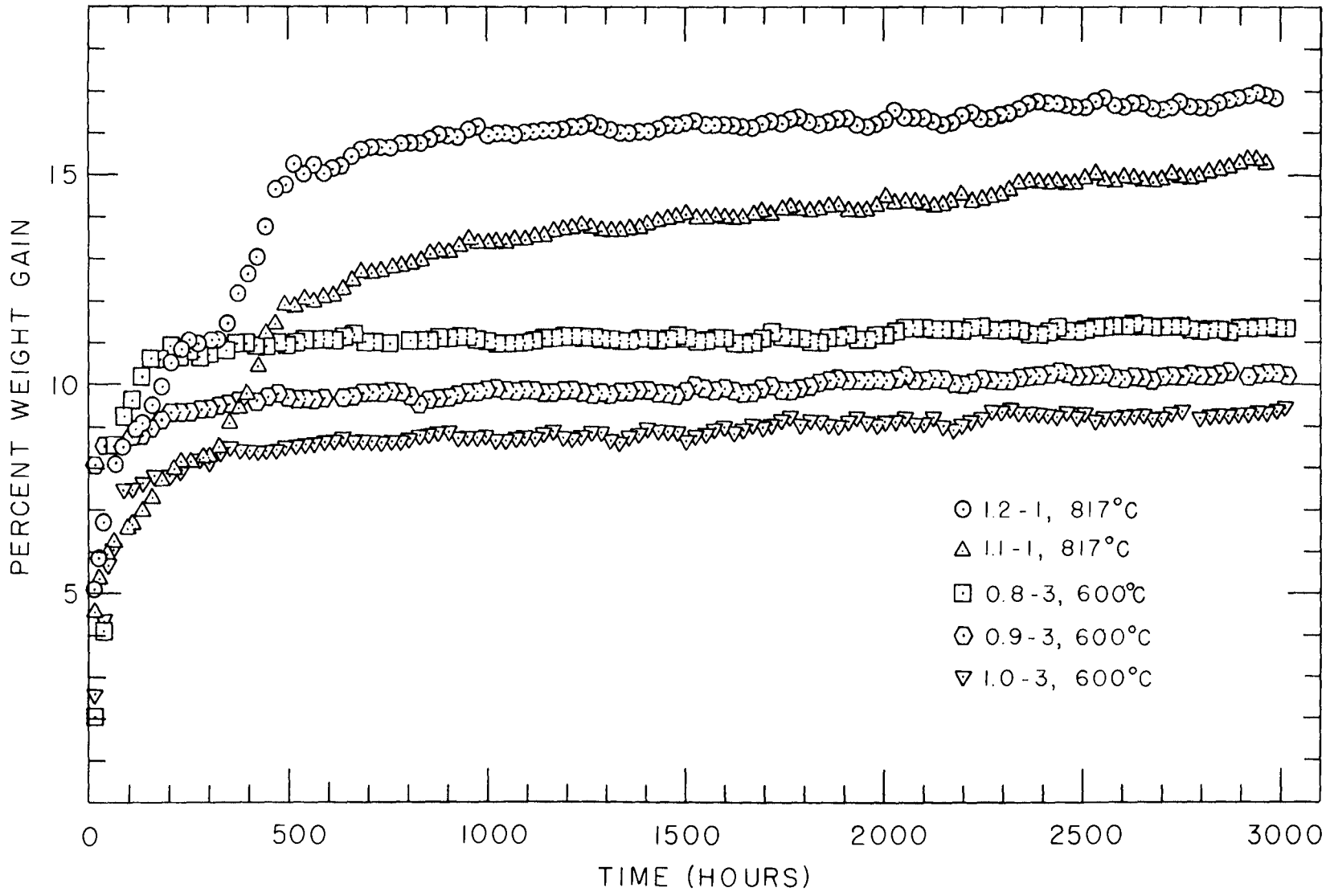

FIGURE 34 Weight gain data for samples 1.2-1, 1.1-1, 0.8-3, 0.9-3, and 1.0-3.

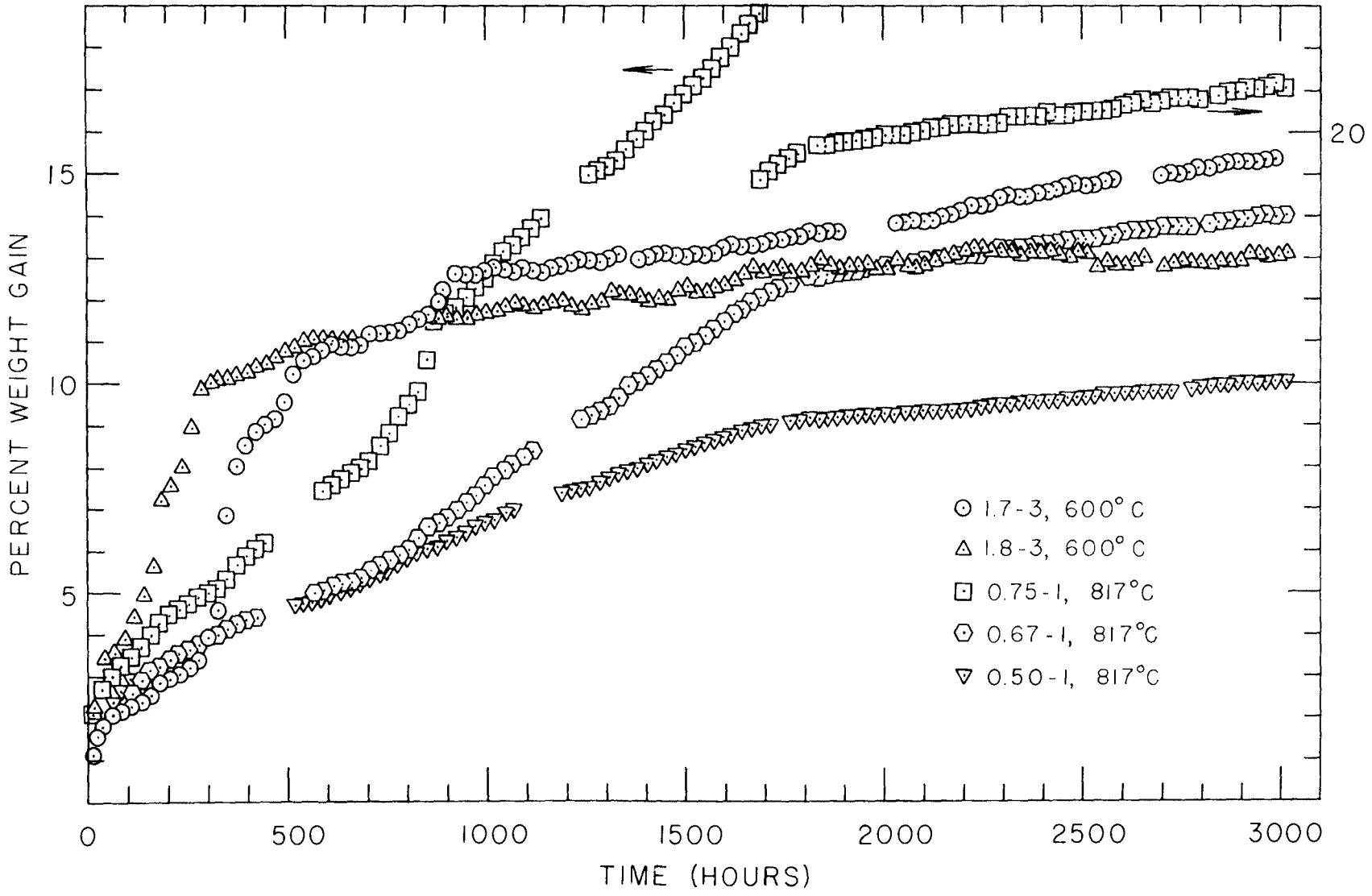

FIGURE 35 Weight gain data for samples 1.7-3, 1.8-3, 0.75-1, 0.67-1, and 0.50-1.

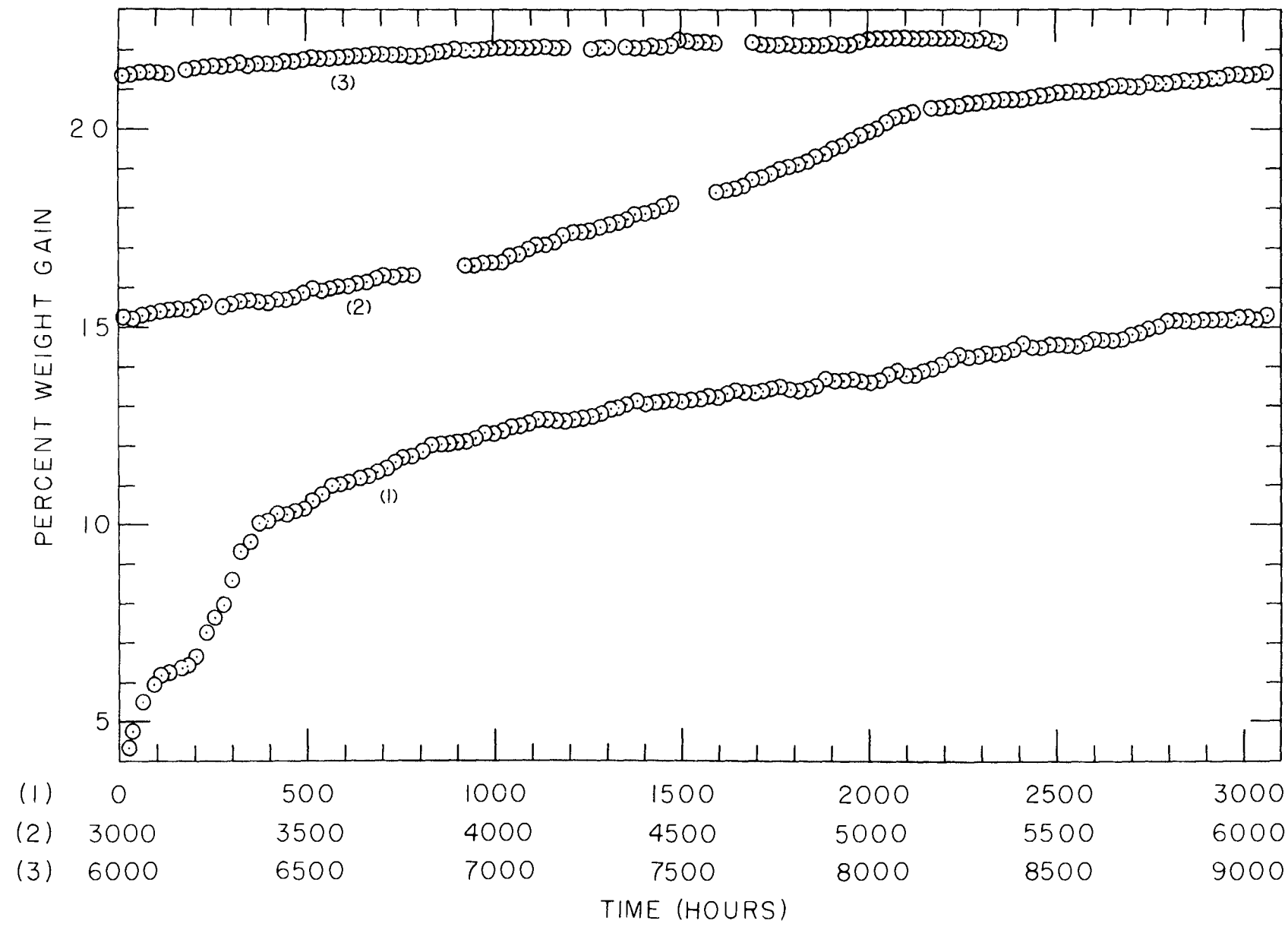

FIGURE 36 Weight gain data for sample 0.9-1.

hours. (The number 1.7 refers to intended Te/Zr mole ratio; the number 2 is the sample number of that composition.) Both weight losses were associated with the arrival of cold, dry weather in early winter. Samples 1.6-2 and 1.3-1, in Fig. 32, and 1.5-3, in Fig. 33, also showed sudden weight losses on the same date but at earlier times, respectively, since these samples were opened after sample 1.7-2 was opened. Throughout the study, all samples showed larger weight gains on rainy days than on dry days. Samples opened in the winter began to gain weight more rapidly in spring near the end of the 300 hour period, as relative humidity increased. For this reason, one sample, 0.9-1 (Fig. 36), opened in the winter, was weighed daily for 8351 hours, about 11 months. In the following fall season when drier weather returned, this sample began to lose weight on extremely dry days. On the basis of the above observations, the gain in weight of the samples is more logically explained as pickup of water rather than oxygen, or even CO_2.

The samples showed which compositions had free, or partially reacted, Zr present. Free Zr was not readily distinguished when the sample was first opened, although the presence of a harder phase was obvious on grinding. There was no free Zr in the first set of samples fired at 817°C; it was observed first in samples with an intended Te/Zr mole ratio of 1.3 or lower fired at 600°C.

X-ray patterns were made of the samples after 3000 hours of exposure to air. There were no sharp lines in the

patterns, but a low, broad band in the range of the (002) and (011) lines (26° to 30° 2θ). The materials were black powders, some with free Zr.

At the start of the hydration studies, it was hypothesized that the samples having higher Te contents would absorb larger amounts of water because of their higher defect content, caused by Zr vacancies. Chemical analysis results provided an opportunity to test this hypothesis, since the amount of weight gained by each sample during exposure to air was also known. Table XII summarizes the chemical analysis results and the respective percent weight gain values. The weight gain values were selected at a constant elapsed time, where possible for a given firing schedule, to provide some degree of reasonable comparison. At the time of these measurements, samples were being opened at the rate of 4 or 5 a week at a time when the weather was uniformly humid. Comparison of samples within a given firing schedule should therefore have some validity.

It can be seen from Table XII that there was no direct relationship between percent weight gained and Te content. The percent weight gained values were converted to moles of water absorbed values, as shown in Appendix J. These values are also tabulated in Table XII and it can be seen there is no direct relationship between moles of water absorbed and Te content. Two explanations for the lack of agreement can be offered. 1) The defect content of the samples was not directly related to the Te content. 2) Choosing the percent

TABLE XII

Comparison of Te Content of Samples to the Percent Weight Gained and Moles of Water Gained During Exposure to Ambient Conditions

Sample	Analyzed Te/Zr Mole Ratio	Percent Weight Gain	Moles of Water Gained	Elapsed Time, Hours
This series was fired at 600°C for 130 hours and air-quenched.				
2.0-6	2.02	16.18	3.135	229
1.9-4	1.70	16.08	2.751	230
1.6-4	1.67	17.21	2.907	230
1.5-1s	1.62	20.71	3.425	228
1.5-1ℓ	1.53	24.38	3.877	228
1.8-6	1.43	15.79	2.399	228
1.3-7	1.37	17.30	2.544	228
1.4-7	1.35	16.73	2.446	229
1.0-8	1.33	16.51	2.391	122
1.1-7	1.29	17.33	2.462	229
1.7-6	1.19	16.67	2.250	229
1.2-10	1.14	16.73	2.198	228
0.9-7	0.94	13.69	1.605	110
0.8-7	0.67	11.65	0.553	121
This series was fired at 817°C for 130 hours and air-quenched.				
2.0-4	2.12	2.42	0.486	84
1.8-4	1.91	11.67	2.168	112
1.9-6	1.86	6.48	1.182	120
1.6-6s	1.69	19.75	3.363	91
1.7-4s	1.65	17.46	2.924	91
1.7-4ℓ	1.47	4.59	0.7100	122
1.5-5ℓ	1.39	18.41	2.745	109
1.6-6ℓ	1.30	12.26	1.750	140

TABLE XII (Cont.)

Sample	Analyzed Te/Zr Mole Ratio	Percent Weight Gain	Moles of Water Gained	Elapsed Time, Hours
This series was fired at 817°C for 130 hours and air-quenched.				
1.4-2	1.52	21.90	3.466	373
1.0-2	1.11	19.25	2.488	361
1.1-2	1.05	22.89	2.861	373
0.9-2	0.92	22.78	2.638	373
1.2-2	0.82	22.41	2.436	373
1.3-2	0.78	19.15	2.027	374
0.8-2	0.72	15.20	1.545	370
This series was fired at 817°C for 130 hours, furnace-cooled to 600°C and soaked for 130 hours, and air-quenched.				
1.0-4	1.32	19.49	2.809	446
1.4-4	0.95	23.42	2.762	446
0.8-4	0.84	19.63	2.162	445
0.9-4	0.82	23.07	2.508	445
1.2-4	0.68	20.81	2.056	443
1.1-4	0.62	19.38	1.832	451
This series was fired at 650°C for 500 hours and air-quenched.				
1.4-6	1.96	20.41	3.867	85
1.3-6	1.42	18.67	2.823	86
1.2-6	1.29	15.87	2.254	86
1.1-6	1.02	13.91	1.710	85

TABLE XII (Cont.)

Sample	Analyzed Te/Zr Mole Ratio	Percent Weight Gain	Moles of Water Gained	Elapsed Time, Hours
This series was fired at 700°C for 200 hours and air-quenched.				
1.4-5	1.51	20.04	3.158	327
1.1-5	1.22	18.26	2.503	323
1.3-5	1.15	18.52	2.446	329
1.2-5	0.98	17.00	2.040	324

weight gained values at almost constant elapsed time did not really eliminate differences in the time-humidity relationships of the different samples. The first reason seems highly likely; in the discussion on relative intensities, in Section IV-C, it was shown that the experimental results could be explained by several values of Te vacancy concentration.

Two series of X-ray samples, 0.8-2 to 1.4-2 (fired at 817°C for 130 hours) and 0.8-4 to 1.4-4 (fired at 817°C for 130 hours, followed by 130 hours at 600°C), were exposed to atmospheric conditions, using the chemical analysis sample to determine the amount of water absorbed. Slow scans were made frequently on these samples to see if new lines were formed during water absorption. No new lines were observed, only a decrease in intensity and broadening of line widths until the peaks were gone. It is interesting to compare these results with the results for samples stored in vials reported in the previous section. Samples fired at 817°C and stored in sealed jars for one year after X-raying had no X-ray pattern, while samples fired at 600°C and stored in sealed jars for one year gave a diffraction pattern for Te.

As mentioned before, some of the samples were discovered to be two-phase, with hard, metallic material in the center of the sample. Where feasible, hydration studies were made on both phases. Sample 1.5-4ℓ, the data for which is plotted in Fig. 33 was ground prior to weighing, but

samples 1.7-4ℓ, 1.6-6ℓ, and 1.5-5ℓ, in Table XII, were not ground. The metallic material of the last three samples was separated from the powder sinter and placed in Al crucibles. Initial weight gain and discoloration was slower than that of ground samples, but by the third day, the samples began breaking up into a black powder with a large increase in weight. The metallic material was apparently porous on a microscopic scale.

F. Evidence for Two-Phase Regions

As mentioned earlier, several samples contained two phases, a hard metallic material and the normal powder sinter. Table XIII summarizes these compositions, with chemical analyses. Sample 1.5-1 was fired at 600°C; the others were fired at 817°C.

Two interesting points can be made regarding the data. The first is that the solid phase has the higher Te content. This is interesting because the melting point of Te is 450°C, while that of Zr is 1852°C.[12] One would expect that at the firing temperatures involved, 600° or 817°C, Te would melt and quickly react with the Zr until it had taken up all the Zr possible. Then, any material not taken into solution would be high in Zr. The latter material would be expected to be similar in texture to that of the original filings. That this reasoning is erroneous for this system is indicated by the data in Table XIII; material with a texture most like the Zr filings is the richest in Te. It is also inter-

TABLE XIII

Chemical Analysis Results on

Two-Phase Samples

Sample	Analyzed Te/Zr Mole Ratio	"Average" Composition
1.5-1ℓ	1.53	1.58
" s	1.62	
1.5-5ℓ	1.39	----
" s	----	
1.6-6ℓ	1.30	1.50
" s	1.69	
1.7-4ℓ	1.47	1.56
" s	1.65	

esting that the metallic phase is distributed along the center of the samples, fired in vertical positions. The second point is that the composition obtained by averaging the chemical analyses of both phases was approximately $ZrTe_{1.55}$. This is a rough approximation however, because relative amounts could not be readily determined.

X-ray patterns of metallic phases were run on two samples, having intended compositions of $ZrTe_{1.5}$ and $ZrTe_{1.7}$, respectively, fired at 600°C for 130 hours. X-ray patterns, summarized in Appendix C, showed that the metallic phases had the basic pattern of $ZrTe_2$ plus many extra lines.

Another two phase region was detected with X-rays at the low Te region, with Te/Zr mole ratios of 1.3 or less. A change in the X-ray pattern from that of a basic $ZrTe_2$ pattern was first noticed with a $ZrTe_{1.1}$ sample (intended composition), fired at 817°C. Many additional lines were observed. All of the lines were broader and less intense than those in 817°C samples having higher Te/Zr mole ratios. All subsequent 817°C samples, from black sample tubes, in this series with lower intended Te/Zr mole ratios showed the same phenomena. X-ray data are summarized in Appendix B. Corresponding samples fired at 600°C, summarized in Appendix C, did not show the same phenomena; diffraction patterns indicated a highly crystalline material. Extra lines were attributed to Zr. The hydration samples of these compositions also had free, or partially reacted, Zr.

Because of differences in X-ray results from corresponding samples fired at 600° and 817°C, another set of samples (0.8-2 to 1.4-2) was fired at 817°C for 130 hours and a second set (0.8-4 to 1.4-4) was fired at 817°C for 130 hours, then at 600°C for 130 hours. Black sample tubes were obtained in both cases. X-ray results from these two sets of samples are summarized in Appendices D and F, respectively. Because the x-ray patterns of the sample set 0.8-2 to 1.4-2 did not reproduce the results obtained from the first set of samples fired at 817°C, chemical analyses were obtained. The results are included with the X-ray data in the Appendices. The X-ray patterns for samples from both of these firings, i.e., the second 817°C firing and the 817°C - 600°C firing, all exhibited many extra lines, some of which were not reproducible from sample to sample. These extra lines could not be reasonably accounted for by Zr, Te, or $ZrTe_3O_8$. Some of the extra lines did, however, match those of hexagonal Zr_4Te_3, found by Hahn and Ness[4] and summarized in Table I. Compositions of these samples ranged from $ZrTe_{0.72}$ to $ZrTe_{1.52}$ for the samples fired at 817°C and from $ZrTe_{0.62}$ to $ZrTe_{1.32}$ for the 817° - 600°C samples.

At this point a tentative association was made between the presence of a second phase and the presence of black coatings on the sample tubes. Different firing procedures were attempted to improve crystallinity and to avoid formation of the black coating on the sample tubes. Samples

were fired for 200 hours at 700°C and 500 hours at 650°C; all yielded black sample tubes. X-ray and chemical analysis results are given in Appendices G and H, respectively. For the 700°C samples, many extra lines were present in samples with analyzed Te/Zr mole ratios of 0.98 to 1.22. Three samples in this series, 0.8-5, 0.9-5, and 1.0-5, burned spontaneously when opened in air. Because of this, three of the samples fired at 650°C, 0.8-6, 0.9-6, and 1.0-6, were opened inside a glove bag under a N_2 atmosphere. The X-ray samples were made inside the glove bag. There were many extra lines in these 650°C samples, with compositions ranging from $ZrTe_{0.87}$ to $ZrTe_{1.96}$; $ZrTe_{1.29}$ was an exception with only two extra lines.

An attempt was also made to produce good samples in the region $ZrTe_{0.5}$ to $ZrTe_{0.75}$. This was the Zr_3Te_2 phase field reported by Hahn and Ness.[4] Samples were fired at 600°C for 145 hours, opened inside the glove bag, ground, resealed under vacuum, and fired for 500 additional hours at 600°C. These samples tubes were clear. X-ray results for these samples are summarized in Appendix I. Samples 0.75-2 ($ZrTe_{0.83}$) and 0.67-2 ($ZrTe_{0.69}$) exhibited the $ZrTe_2$ pattern with some extra lines; the lines were broad. Sample 0.50 ($ZrTe_{0.21}$) had a different pattern, near that of Zr, with some extra lines. Free Zr was observed in the chemical analysis samples of 0.50-2 and 0.67-2. An earlier run of samples 0.75-1, 0.67-1, and 0.50-1, fired at 817°C for 130 hours, also failed to produce single-phase Zr_3Te_2 samples.

X-ray results for these samples are summarized in Appendix B.

To summarize, for all heating conditions, X-ray patterns of samples in the composition range $ZrTe_{0.62}$ - $ZrTe_{1.3}$ were combinations of ZrTe or $ZrTe_2$ lines, possible superlattice lines, and lines that matched those of the hexagonal $ZrTe_{0.75}$ structure reported by Hahn and Ness.[4] Most of the samples fired at temperatures lower than 817°C had free, or partially reacted, Zr. More work needs to be done to delineate the phases present in this region. Longer firing times will be necessary to react the Zr thoroughly.

All samples were air-quenched. Hahn and Ness[4] make no statement as to which cooling method they employed. The inability to reproduce their results in the low Te region, i.e., inability to obtain the single phase Zr_3Te_2 solid solution region, raises the question as to whether they may have cooled their samples very slowly. Hahn and Ness[4] reported that ZrTe was thermally unstable at 900°C, decomposing to $ZrTe_2$ and Zr_4Te_3, yet x-ray patterns of samples in the composition range $ZrTe_{0.8}$ - $ZrTe_{1.3}$ did not indicate this. The present work on quenched samples does indicate, however, existence of a two-phase region in the composition range $ZrTe_{0.62}$ - $ZrTe_{1.3}$.

G. Reactions With Organic Liquids

Treatment of $ZrTe_2$ with pyridine and toluene was undertaken to see if $ZrTe_2$ would form intercalation compounds

with organic liquids similar to those formed by TaS_2.[13] TaS_2 has the same structure as $ZrTe_2$, i.e., the CdI_2 structure.[2]

$ZrTe_2$ (sample 2.0-4) was treated with toluene and pyridine for 10 days in air. The material changed color from golden-brown to black while immersed in the liquids. Aggregates of material were somewhat difficult to crush after immersion, but subsequent grinding of the material was easy.

X-ray patterns of these materials, starting at 3° 2θ with CuKα radiation (29 Å.), exhibited no lines other than those of $ZrTe_2$. Intensities were somewhat lower than those observed for corresponding lines in untreated samples.

Though no intercalation compounds were formed by $ZrTe_2$ in these experiments, it should not be assumed that no intercalation compounds will form. Later information on the intercalation complexes of TaS_2[14] and other sulfides showed that temperatures as high as 200°C and reaction times of up to 30 days were required to form complexes. Furthermore, the $ZrTe_2$ was not ground before being placed in the organic liquids. Grinding might aid penetration of the lattice by the liquids by breaking up the aggregate into smaller plates.

V. CONCLUSIONS

On the basis of theoretical and experimental relative intensities, a structural model of 15% Zr vacancies at z = 0 and Te vacancy concentrations of 0 to 5% is proposed for compositions in the range $ZrTe_{1.5}$ - $ZrTe_2$, synthesized between 600°C and 817°C. The best structural model that can be proposed at present for compositions in the range ZrTe - $ZrTe_{1.5}$, synthesized between 600°C and 817°C, is one in which the number of Zr vacancies located at z = 0 ranges from 0% to 30%, with the concentration of Te vacancies ranging from 0 to 35%. More experimental measurements are required before the defect structures in the composition range ZrTe - $ZrTe_{1.5}$ can be completely delineated. Because there is considerable overlap of theoretical relative intensities for different values of the Zr disorder parameter and the Te vacancy concentration, density measurements appear to offer the most promise for clarification of the defect structures in this region.

Only ordered structures were formed by quenching from temperatures between 600° and 817°C. Samples fired at higher temperatures should be investigated to determine if order-disorder transformation of the tellurides occur.

X-ray results indicate existence of a two phase region in the composition range $ZrTe_{0.7}$ - $ZrTe_{1.3}$ between 650° and 817°C. The phases are hexagonal Zr_4Te_3, first reported by Hahn and Ness,[4] and either a ZrTe or $ZrTe_2$ superlattice.

More work needs to be done to clarify the structures of the compositions in this region. Reactivity of the components is a problem for these compositions. This can be improved by the use of Zr which has been hydrided and subsequently dissociated. Since this form of Zr is very fine and therefore pyrophoric, formulation of compositions must be done in an inert atmosphere.

Two phases were also observed visually to have formed at 600° and 817°C. The mean composition of the two phases is approximately $ZrTe_{1.55}$.

Hydration studies indicate that the tellurides react with water, rather than oxygen, upon exposure to the atmosphere. The rate of weight gain of these samples is related to relative humidity.

To insure that extra lines observed in X-ray patterns of many samples are characteristic of the telluride rather than of a hydrate, all X-ray samples in future work should be prepared in a dry environment.

The tellurides corrode Pt when placed directly into a furnace at 870°C. This phenomenon did not occur with tellurides which were preoxidized at 600°C, suggesting that the vapor species above unoxidized tellurides are highly corrosive. A Te analogue of PtO_2 might be responsible for vaporization of the Pt crucible.

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VITA

Frances Dolores Jenkins, nee Gaidos, was born on January 25, 1938, in Oakland, California. She received her primary and secondary education in Berkeley, California. She received her college education from the University of California, in Berkeley, California, where she received a Bachelor of Science degree in Ceramic Engineering in January 1960 and a Master of Science degree in Ceramic Engineering in August 1961. She was employed by American Glass Research, Inc., in Butler, Pennsylvania, from September 1961 to August 1965.

She has been enrolled in the Graduate School of the University of Missouri-Rolla since September 1965. She was employed as a Instructor in the Department of Ceramic Engineering from 1965 to 1970.

APPENDIX A

COMPUTER PROGRAMS

1. Section Common to All Relative Intensity Programs

This section appears in every program in which relative intensities are calculated. It will be referred to as the COMMON SECTION in each program. In this section, values of the Miller indices of the planes of interest, the 2θ value of the diffraction peak of each plane, the multiplicity of each plane, and the constants used in the atomic scattering factor calculation are read into the computer. The calculations made are the Lorentz-polarization factor and the atomic scattering factors for each value of 2θ.

```
C
C     N STANDS FOR THE NUMBER OF LINES TO BE READ, A
C     CHANGEABLE VALUE.  MAXIMUM VALUE IS 50.
      N = 10
C     THIS DO LOOP READS IN THE HKL VALUE, THE 2-THETA
C     VALUE, AND THE MULTIPLICITY OF EACH LINE BEING CON-
C     SIDERED.
      DO 1000 I = 1,N
      READ (1,1100) H(I), K(I), L(I), THET2(I), MULT(I)
 1100 FORMAT  (3D10.1, D10.2, D10.1)
 1000 CONTINUE
C
C     THIS DO LOOP PRINTS THE VALUES READ IN FOR INITIAL
C     VALUE CHECK.
      DO 1001 I = 1,N
      WRITE (3,1101) I, H(I), K(I), L(I), THET2(I), MULT(I)
 1101 FORMAT (3X, 'THESE VALUES HAVE BEEN READ IN FOR THE DI
     1FFRACTION PEAKS BEING CONSIDERED',//(3X, 'I =', 2X, 'H
     2 =', 2X, F3.1, 2X, 'K =', 2X, F3.1, 2X, 'L =', 2X, '2-
     3THETA IN DEGREES =', 2X, F6.2, 2X, 'MULTIPLICITY =',
     42X, F4.1))
 1001 CONTINUE
C
C     THESE NEXT STATEMENTS READ IN THE CONSTANTS FOR CALCU-
C     LATING THE ATOMIC SCATTERING FACTORS.  CA = CATION,
C     AN = ANION
      READ (1,1200) (CAA(I),I=1,4)
      READ (1,1200) (CAB(I),I=1,4)
      READ (1,1200) (ANA(I),I=1,4)
      READ (1,1200) (ANB(I),I=1,4)
```

```
 1200 FORMAT (4D15.5)
      READ (1,1201) CAC, ANC
 1201 FORMAT (2D15.5)
C
C     THIS SECTION PRINTS THE CONSTANTS THAT ARE USED FOR
C     CALCULATING ATOMIC SCATTERING FACTORS.
      WRITE (3,1202) (CAA(I),I=1,4)
 1202 FORMAT (//3X, 'CATION A VALUES ARE', 4F10.5)
      WRITE (3,1203) (CAB(I),I=1,4)
 1203 FORMAT (//3X, 'CATION B VALUES ARE', 4F10.5)
      WRITE (3,1204) (ANA(I),I=1,4)
 1204 FORMAT (//3X, 'ANION A VALUES ARE', 4F10.5)
      WRITE (3,1205) (ANB(I),I=1,4)
 1205 (//3X, 'ANION B VALUES ARE', 4F10.5)
      WRITE (3,1206) CAC, ANC
 1206 FORMAT (//3X, 'CATION C VALUE IS', 2X, F1015, 2X, 'ANI
     1ON C VALUE IS', 2X, F10.5)
C
C     THIS NEXT SECTION CONVERTS THE 2-THETA VALUES INTO RA-
C     DIANS AND STORES THESE VALUES.
      TWOPI = 6.283185307179586
      DO 1300 I=1,N
      THET2R(I) = TWOPI*THET2(I)/36010D0
 1300 CONTINUE
C
C     THIS NEXT SECTION PRINTS THE 2-THETA VALUES IN DE-
C     GREES AND RADIANS.
      DO 1301 I=1,N
      WRITE (3,1302) THET2(I), THET2R(I)
 1302 FORMAT (//(3X, 'FOR 2-THETA IN DEGREES OF', F8.2, 2X,
     1'IN RADIANS IT IS', F12.4))
 1301 CONTINUE
C
C     THIS NEXT SECTION CALCULATES AND STORES THE LORENTZ-
C     POLARIZATION FACTOR, POLFAC, FOR THE NUMBER OF LINES
C     UNDER CONSIDERATION.
      DO 1400 I=1,N
      POLFAC(I) = (1.0D0 + DCOS(THET2R(I))**2)/(DSIN(THET2R(
     1I)/2.0D0**2*DCOS(THET2R(I)/2.0D0))
      WRITE (3,1401) THET2(I), POLFAC(I)
 1401 FORMAT (//10X, 'FOR 2-THETA IN DEGREES OF', F8.2, 2X,
     2'LORENTZ-POLARIZATION FACTOR IS', F28.16)
 1400 CONTINUE
C
C     THIS SECTION CALCULATES AND STORES THE ATOMIC SCATTER-
C     ING FACTORS.  THE WAVELENGTH LAMDA IS A CHANGEABLE
C     VALUE.
      LAMDA = 1.54178D0
      DO 5000 I=1,N
      FAC = DSIN(THET2R(I)/2.0D0)**2/(LAMDA*LAMDA)
C
C     THIS NEXT SECTION CALCULATES THE ATOMIC SCATTERING
C     FACTOR FOR THE CATION.
```

```
      ATOMCA(I) = CAA(1)*DEXP((-CAB(1))*FAC) + CAA(2)*DEXP((
     1-CAB(2))*FAC + CAA(3)*DEXP((-CAB(3))*FAC) + CAA(4)*DEX
     2P((-CAB(4))*FAC) + CAC
C
C     THIS NEXT SECTION CALCULATES THE ATOMIC SCATTERING
C     FACTOR FOR THE ANION.
      ATOMAN(I) = ANA(1)*DEXP((-ANB(1))*FAC) + ANA(2)*DEXP((
     1-ANB(2))*FAC) + ANA(3)*DEXP((-ANB(3))*FAC) + ANA(4)*DE
     2XP((-ANB(4))*FAC) + ANC
C     NOW WE PRINT THE RESULT.
      WRITE (3,5001) THET2R(I), ATOMCA(I), ATOMAN(I)
 5001 FORMAT (//3X, 'FOR 2-THETA (RAD.) =', F12.8, 2X, 'THE
     1ATOMIC SCATTERING FACTOR FOR THE CATION =', F12.8, 2X,
     2'AND FOR THE ANION =', F12.8)
 5000 CONTINUE
C
```

2. Relative Intensity Calculation for Variable Te Coordinate in $ZrTe_2$ Structure

This program calculates relative intensities for (hkl) lines as a function of u, the variable Te coordinate.

```
C     THIS PROGRAM COMPUTES RELATIVE INTENSITIES FOR ANY
C     MATERIAL HAVING THE CADMIUM IODIDE STRUCTURE.  THERE
C     IS ONE FUNCTION SUBPROGRAM TO CALCULATE THE STRUCTURE
C     FACTOR.
      IMPLICIT REAL*8 (A-F,O-Z)
      DOUBLE PRECISION H,K,L, MULT, INTENS, LAMDA
      DIMENSION H(50), K(50), L(50), THET2(50), MULT(50),
     1THET2R(50), INTENS(50), RELINT(50), U(50), POLFAC(50),
     2CAA(4), CAB(4), ANA(4), ANB(4), ATOMCA(50), ATOMAN(50)
                              .
                              .
                              .
                      COMMON SECTION
                              .
                              .
                              .
C
C     THIS NEXT SECTION CALCULATES THE INTENSITY VALUES OF
C     EACH LINE.  THIS WILL BE A NESTED DO LOOP BECAUSE WE
C     WANT TO CALCULATE INTENSITIES FOR DIFFERENT VALUES OF
C     THE Z-PARAMETER.  IN ADDITION, THE INTENSITY CALCULA-
C     TION IS DIVIDED INTO TWO SECTIONS BECAUSE TWO GEOME-
C     TRIC STRUCTURE FACTORS HAVE TO BE CALCULATED FOR ALL
C     HKL AND H0L LINES.  THIS IS THE FIRST INTENSITY DO
C     LOOP.
      READ (1,2001) (U(J),J=1,20)
 2001 FORMAT (5D10.2)
C     U(J) IS THE VARIABLE Z-PARAMETER.
```

```
      WRITE (3,2002) (U(J),J=1,20)
 2002 FORMAT (//8X, 'THE VALUES FOR THE Z-PARAMETER ARE'/(3X
     1, 10F10.3))
      DO 2000 J=1,20
      B = U(J)
      N1 = 4
C     THE VALUE N1 SETS THE NUMBER OF LINES THAT HAVE A
C     DOUBLE STRUCTURE FACTOR CALCULATION.  THIS VALUE IS
C     CHANGEABLE.
      DO 2100 I=1,N1
      INTENS(I) = MULT(I)*POLFAC(I)*STRFAC(H(I),K(I),L(I),B,
     1THET2R(I),ATOMCA(I),ATOMAN(I))
 2100 CONTINUE
C
C     THIS IS THE SECTION FOR SINGLE STRUCTURE FACTOR CALCU-
C     LATION.
      NPLUS = N1 + 1
      DO 2200 I=NPLUS,N
      INTENS(I) = MULT(I)*POLFAC(I)*STRFAK(H(I),K(I),L(I),B,
     1ATOMCA(I),ATOMAN(I))
 2200 CONTINUE
C
C     NOW WE CALCULATE THE RELATIVE INTENSITY VALUES.  MY
C     MOST INTENSE PEAK IS THE FIRST PEAK.
      DO 2700 I=1,N
      RELINT(I) = INTENS(I)/INTENS(1)
 2700 CONTINUE
C
C     NOW WE PRINT FOR THE PRESENT Z-PARAMETER THE HKL VA-
C     LUES WITH THE  INTENSITY AND RELATIVE INTENSITY
C     VALUES.
      WRITE (3,2301) B
 2301 FORMAT(///20X,'THE Z COORDINATE IS', F8.3)
      DO 2303 I=1,N
      WRITE (3,2302) H(I), K(I), L(I), INTENS(I), RELINT(I)
 2302 FORMAT (//3X, 'THE HKL VALUE IS', 3F5.1, 3X, 'THE INT
     1ENSITY IS', F33.16, 3X, 'THE RELATIVE INTENSITY IS',
     2F8.4)
 2303 CONTINUE
C
C     NOW TO GO BACK AND REDO FOR THE NEXT VALUE OF THE Z-
C     PARAMETER.
 2000 CONTINUE
C
C     THE MAIN PROGRAM IS COMPLETED.
      STOP
      END

      FUNCTION STRFAC (HD,KD,LD,UD,THET2D,ATOMCD,ATOMAD)
      IMPLICIT REAL*8 (A-Z)
C
C     THIS SUBPROGRAM CALCULATES THE STRUCTURE FACTOR.
C     THE VALUES TRANSFERRED IN ARE H, K, L, THET2R, THE
```

```
C      Z-PARAMETER, AND THE TWO ATOMIC SCATTERING FACTORS.
C
C      THIS SECTION CHECKS THE VALUES TRANSFERRED.
       WRITE (3,4000) HD, KD, LD, UD, THET2D, ATOMCD, ATOMAD
 4000 FORMAT (//15X, 'THESE VALUES HAVE BEEN TRANSFERRED FRO
      1M THE MAIN PROGRAM'/5X, 'H =', F4.1, 2X, 'K =', F4.1,
      22X, 'Z =', F6.3, 2X, '2-THETA (RAD.) =', F12.8, 2X, 'C
      3ATION ATFAC =', F12.8, 2X, 'ANION ATFAC =', F12.8)
C
C      THIS SECTION IS THE OPTIONAL STRUCTURE FACTOR CALCULA-
C      TION.
       TWOPI = 6.283185307179586
       STRF1C = ATOMCD + ATOMAD*(DCOS(TWOPI*(KD/3.0D0 + 2.0D0
      1*HD/3.0D0 + LD*UD)) + DCOS(TWOPI*(2.0D0*KD/3.0D0 + HD/
      23.0D0 + LD*(0UD))))
       STRFAC = STRF1C*STRF1C
       GO TO 4500
C
C      THIS IS THE ENTRY POINT WHEN ONLY ONE STRUCTURE FAC-
C      TOR IS CALC'D.
       ENTRY STRFAK (HD,KD,LD,UD,THET2D,ATOMCD,ATOMAD)
       WRITE (3,4000) HD, KD, LD, UD, THET2D, ATOMCD, ATOMAD
       STRFAC = 0.0D0
 4500 STRF2C = ATOMCD + ATOMAD*(DCOS(TWOPI*(HD/3.0D0 + 2.0D0
      1*KD/3.0D0 + LD*UD)) + DCOS(TWOPI*(2.0D0*HD/3.0D0 + KD/
      23.0D0 + LD*(0&D))))
       STRFAC = STRFAC + STRF2C*STRF2C
       WRITE (3, 4501) STRFAC
 4501 FORMAT (3X, "THE CALCULATED STRUCTURE FACTOR IS', F28.
      116)
       RETURN
```

3. Relative Intensity Calculation for Degrees of Zr Vacancy Disorder

This program calculates relative intensities for (hkl) lines as a function of the degree of disorder of Zr vacancies.

```
C      THIS PROGRAM CALCULATES RELATIVE INTENSITIES FOR COM-
C      POSITIONS BETWEEN ZRTE AND ZRTE2 (NICKEL ARSENIDE AND
C      CADMIUM IODIDE STRUCTURES, RESPECTIVELY) FOR DIFFERENT
C      DEGREES OF ORDERING.  A COMPLETELY RANDOM STRUCTURE
C      HAS 50% OF THE POSSIBLE VOIDS ON EACH ZR LAYER.
       IMPLICIT REAL*8 (A-F,O-Z)
       DOUBLE PRECISION H, K, L, MULT, INTENS, LAMDA
       DIMENSION H(50), K(50), L(50), THET2(50), MULT(50),
      1THET2R(50), INTENS(50), RELINT(50), POLFAC(50), CAA(4)
      2, CAB(4), ANA(4), ANB(4), ATOMCA(50), ATOMAN(50), VF(2
      30), ORPAR(10)
C
```

```
                              .
                              .
                              .
                        COMMON SECTION
                              .
                              .
                              .
C     THIS NEXT SECTION CALCULATES THE INTENSITIES OF EACH
C     LINE.  THIS WILL BE A NESTED DO LOOP, ONE LOOP FOR
C     THE FRACTION OF VOIDS FOR A GIVEN COMPOSITION AND THE
C     OTHER LOOP FOR THE DEGREE OF ORDERING.  IN ADDITION
C     THI INTENSITY CALCULATION IS DIVIDED INTO TWO SECTIONS
C     BECAUSE TWO GEOMETRIC STRUCTURE FACTORS HAVE TO BE
C     CALCULATED FOR ALL HKL AND H0L LINES.  THIS IS THE
C     FIRST INTENSITY DO LOOP.
C
C     IT SHOULD BE NOTED THAT THE MULTIPLICITY VALUES FOR
C     THE CADMIUM IODIDE STRUCTURE ARE BEING USED WITH THE
C     ATOMIC POSITIONS FOR THE NICKEL ARSENIDE STRUCTURE.
C
C     READ IN THE FRACTION VOID VALUES, VF, AND PRINT FOR
C     CHECK.
      READ (1,2001) (VF(J), J=1,10)
 2001 FORMAT (5D10.2)
      WRITE (3,2002) (VF(J), J=1,10)
 2002 FORMAT (/8X, 'THE FRACTION VOID VALUES ARE:'/3X, 10F8.
     12)
C
C     READ IN THE ORDER PARAMETERS, ORPAR, AND PRINT FOR
C     CHECK.
      READ (1,2003) (ORPAR(M), M=1,6)
 2003 FORMAT (6D10.1)
      WRITE (3,2004) (ORPAR(M), M=1,6)
 2004 FORMAT (/8X, 'THE VALUES OF THE ORDER PARAMETER ARE:',
     12X, 6F5.2)
C
C     START THE DO LOOPS
      DO 2100 J = 1,10
C
      DO 2200 M = 1,6
C     THE VALUE N1 SETS THE NUMBER OF LINES THAT HAVE A
C     DOUBLE STRUCTURE FACTOR CALCULATION.  THIS VALUE IS
C     CHANGEABLE.
      N1 = 4
      DO 2202 I = 1,N1
      INTENS(I) = MULT(I)*POLFAC(I)*STRFAC(H(I),K(I),L(I),VF
     1(J),ORPAR(M),THET2R(I),ATOMCA(I),ATOMAN(I))
 2202 CONTINUE
C
C     THIS IS THE SECTION FOR SINGLE STRUCTURE FACTOR CALCL-
C     LATION.
      NPLUS = N1 + 1
      DO 2203 I = NPLUS,N
```

```
      INTENS(I) = MULT(I)*POLFAC(I)*STRFAK(H(I),K(I),I(I),VF
     1(J),ORPAR(M),THET2R(I),ATOMCA(I),ATOMAN(I))
 2203 CONTINUE
C
C     NOW WE CALCULATE THE RELATIVE INTENSITY VALUES.  MY
C     MOST INTENSE PEAK IS THE FIRST PEAK.
      DO 2700 I=1,N
      RELINT(I) = INTENS(I)/INTENS(1)
 2700 CONTINUE
C
C     NOW WE PRINT THE HKL VALUE, THE INTENSITY, AND THE
C     RLATIVE INTENSITY FOR A GIVEN VOID FRACTION AND ALL
C     ORDER PARAMETERS.
C
      WRITE (3,2251) VF(J), ORPAR(M)
 2251 FORMAT (//15X, 'THE VOID FRACTION IS', F4.2, 'AND THE
     1ORDER PARAMETER IS', F4.2)
      DO 2250 I = 1,N
      WRITE (3,2252) H(I), K(I), L(I), INTENS(I), RELINT(I)
 2252 FORMAT (//3X, 'HKL =', 3F5.1, 'THE INTENSITY IS', F31.
     116, 3X, 'THE RELATIVE INTENSITY IS', F8.4)
 2250 CONTINUE
C
C     NOW TO GO BACK AND REDO FOR ANOTHER VALUE OF THE
C     ORDER PARAMETER.
 2200 CONTINUE
C
C     NOW TO GO BACK AND REDO FOR ANOTHER VALUE OF VOID
C     FRACTION.
 2100 CONTINUE
C
C     THE MAIN PROGRAM IS COMPLETED.
      STOP
      END

      FUNCTION STRFAC (HD,KD,LD,VFD,ORPARD,THET2D,ATOMCD,ATO
     1AD)
      IMPLICIT REAL*8 (A-Z)
C
C     THIS SUBPROGRAM CALCULATES THE STRUCTURE FACTOR.  THE
C     VALUES TRANSFERRED IN ARE H, K, L, THET2R, THE ORDER-
C     ING PARAMETER, THE FOID FRACTION, AND THE TWO ATOMIC
C     SCATTERING FACTORS.
C
C     THIS SECTION IS THE OPTIONAL STRUCTURE FACTOR
C     CALCULATION.
      TWOPI = 6.283185307179586
      PI = 3.141592653589793
      STRF1C = ATOMCD*((1.0D0 - ORPARD*VFD) + (1.0D0 - VFD +
     1ORPARD*VFD)*DCOS(PI*LD)) + ATOMAD*(DCOS(TWOPI*(KD/3.0D
     120 + 2.0D0*HD/3.0D0 + LD/4.0D0)) + DCOS(TWOPI*(2.0D0*KD
     13/3.0D0 + HD/3.0D0 + 3.0D0*LD/4.0D0)))
      STRFAC = STRF1C*STRF1C
```

```
      GO TO 4500
C
C     THIS IS THE ENTRY POINT WHEN ONLY ONE STRUCTURE FACTOR
C     IS CALC'D.
      ENTRY STRFAK (HD,KD,LD,VFD,ORPARD,THET2D,ATOMCD,ATOMAD)
      STRFAC = 0.0D0
 4500 STRF2C = ATOMCD*((1.0D0 - ORPARD*VFD) + (1.0D0 - VFD +
     1ORPARD*VFD)*DCOS(PI*LD)) + ATOMAD*(DCOS(TWOPI*(HD/3.0D
     20 + 2.0D08KD/3.0D0 + LD/4.0D0)) + DCOS(TWOPI*(2.0D0*HD
     3/3.0D0 + KD/3.0D0 + 3.0D0*LD/4.0D0)))
      STRFAC =STRFAC + STRF2C*STRF2C
      RETURN
      END
```

4. Relative Intensity Calculation for Degrees of Zr Vacancy Disorder and Te Vacancy Concentration

This program calculates relative intensities for (hkl) lines as a function of the degree of disorder of the Zr vacancies, as well as different concentrations of Te vacancies.

```
C     THIS PROGRAM CALCULATES RELATIVE INTENSITIES FOR COM-
C     POSITIONS BETWEEN ZRTE AND ZRTE2 (NICKEL ARSENIDE AND
C     CADMIUM IODIDE STRUCTURES, RESPECTIVELY) FOR DIFFERENT
C     DEGREES OF ORDERING.  A COMPLETELY RANDOM STRUCTURE
C     HAS 50% OF THE POSSIBLE VOIDS ON EACH ZR LAYER.  THIS
C     PROGRAM NOW ALSO CALCULATES RELATIVE INTENSITIES AS A
C     FUNCTION OF NUMBER OF VACANCIES IN THE TE LAYERS.
      IMPLICIT REAL*8 (A-F,O-Z)
      DOUBLE PRECISION H, K, L, MULT, INTENS, LAMDA
      INTEGER Q
      DIMENSION H(50), K(50), L(50), THET2(50), MULT(50),
     1THET2R(50), INTENS(50), RELINT(50), POLFAC(50), CAA(4)
     2, CAB(4), ANA(4), ANB(4), ATOMCA(50), ATOMAN(50), ZRS1
     3(5,11), ZRS2(5,11), TEV(5,11), ORPAR(10), COMP(11)
                          .
                          .
                          .
                    COMMON SECTION
                          .
                          .
                          .
C     THIS NEXT SECTION CALCULATES THE INTENSITIES OF EACH
C     LINE.  THIS WILL BE A NESTED DO LOOP, ONE LOOP FOR THE
C     FRACTION OF VOIDS FOR A GIVEN COMPOSITION AND THE
C     OTHER LOOP FOR THE DEGREE OF ORDERING.  IN ADDITION
C     THE INTENSITY CALCULATION IS DIVIDED INTO TWO SECTIONS
C     BECAUSE TWO GEOMETRIC STRUCTURE FACTORS HAVE TO BE
```

```
C      CALCULATED FOR ALL HKL AND H0L LINES.  THIS IS THE
C      FIRST INTENSITY DO LOOP.
C
C      IT SHOULD BE NOTED THAT THE MULTIPLICITY VALUES FOR
C      THE CADMIUM IODIDE STRUCTURE ARE BEING USED WITH THE
C      ATOMIC POSITIONS FOR THE NICKEL ARSENIDE STRUCTURE.
C
C      READ IN THE ZR OCCUPANCY VALUES FOR THE DIFFERENT
C      COMPOSITIONS AND WITH THE TE VOID CONTENT.  ZRDIF =
C      (ZRS1 - ZRS2).
       READ (1,2001) ((ZRS1(Q,J), ZRS2(Q,J), TEV(Q,J), Q=1,5)
      1,J= 1,11)
 2001 FORMAT (2D10.3, D10.2)
       WRITE (3,2002) ((ZRS1(Q,J), ZRS2(Q,J), TEV(Q,J), Q=1,5
      1),J = 1,11)
 2002 FORMAT (/3X, 'ZR SITE 1 =', F7.3, 2X, 'ZR SITE 2 =',
      1F7.3, 2X, 'TE VOID FRACTION/LAYER =', F6.2)
       READ (1,2005) (COMP(J), J = 1,11)
 2005 FORMAT (6D10.1)
       WRITE (3,2006) (COMP(J), J = 1,11)
 2006 FORMAT (/8X, 'THE TE/ZR MOLE RATIOS ARE:', 2X, 11F6.2)
C
C      READ IN THE ORDER PARAMETERS, ORPAR, AND PRINT FOR
C      CHECK.
       READ (1,2003) (ORPAR(M), M=1,4)
 2003 FORMAT (4D10.1)
       WRITE (3,2004) (ORPAR(M),M=1,4)
 2004 FORMAT (/8X, 'THE VALUES OF THE ORDER PARAMETER ARE:',
      12X, 4F5.2)
C
C
C      START THE DO LOOPS.
       DO 2100 J = 1,11
C
       DO 2200 M = 1,4
C
       DO 2300 Q = 1,5
C      THE VALUE N1 SETS THE NUMBER OF LINES THAT HAVE A
C      DOUBLE STRUCTURE FACTOR CALCULATION.  THIS VALUE IS
C      CHANGEABLE.
       N1 = 4
       DO 2202 I = 1,N1
       INTENS(I) = MULT(I)*POLFAC(I)*STRFAC(H(I),K(I),L(I),TH
      1ET2R(I),ATOMCA(I),ATOMAN(I),ORPAR(M),TEV(Q,J),ZRS1(Q,J
      2),ZRS2(Q,J))
 2202 CONTINUE
C
C      THIS IS THE SECTION FOR SINGLE STRUCTURE FACTOR CAL-
C      CULATION.
       NPLUS = N1 + 1
       DO 2203 I = NPLUS,N
       INTENS(I) = MULT(I)*POLFAC(I)*STRFAK(H(I),K(I),L(I),TH
      2ET2R(I),ATOMCA(I),ATOMAN(I),ORPAR(M),TEV(Q,J),ZRS1(Q,J
```

```
     3),ZRS2(Q,J))
 2203 CONTINUE
C
C      NOW WE CALCULATE THE RELATIVE INTENSITY VALUES.  MY
C      MOST INTENSE PEAK IS THE FIRST PEAK.
       DO 2700 I=1,N
       RELINT(I) = INTENS(I)/INTENS(1)
 2700 CONTINUE
C
C      NOW WE PRINT THE HKL VALUE, THE INTENSITY, AND THE
C      RELATIVE INTENSITY FOR A GIVEN COMP'N, TE OCCUPATION,
C      AND ZR ORDER PARAMETER.
C
       WRITE (3,2251) COMP(J), ORPAR(M), TEV(Q,J)
 2251 FORMAT (//10X, 'THE COMPOSITION IS', F6.2, 3X, 'THE OR
     1DER PARAMETER IS', F6.2, 3X, 'AND THE TE OCCUPATION IS
     2', F6.2)
       DO 2250 I = 1,N
       WRITE (3,2252) H(I), K(I), L(I), INTENS(I), RELINT(I)
 2252 FORMAT (//3X, 'HKL =', 3F5.1, 'THE INTENSITY IS', F31.
     116, 3X, 'THE RELATIVE INTENSITY IS', F8.4)
 2250 CONTINUE
C
C      NOW TO GO BACK AND REDO FOR ANOTHER VALUE OF TE
C      OCCUPANCY.
 2300 CONTINUE
C
C      NOW TO GO BACK AND REDO FOR ANOTHER VALUE OF THE ORDER
C      PARAMETER.
 2200 CONTINUE
C
C      NOW TO GO BACK AND REDO FOR ANOTHER VALUE OF VOID
C      FRACTION.
 2100 CONTINUE
C
C      THE MAIN PROGRAM IS COMPLETED.
       STOP
       END

       FUNCTION STRFAC (HD,KD,LD,THET2D,ATOMCD,ATOMAD,ORPARD,
     1TEVD,ZRS1D,ZRS2D)
       IMPLICIT REAL*8 (A-Z)
C
C      THIS SUBPROGRAM CALCULATES THE STRUCTURE FACTOR.  THE
C      VALUES TRANSFERRED IN ARE H, K, L, THET2R, THE ORDER-
C      ING PARAMETER, THE VOID FRACTION, AND THE TWO ATOMIC
C      SCATTERING FACTORS.
C
C      THIS SECTION IS THE OPTIONAL STRUCTURE FACTOR
C      CALCULATION.
       TWOPI = 6.283185307179586
       PI = 3.141592653589793
       ZRDIFD = ZRS1D - ZRS2D
```

```
      STRF1C = ATOMCD*((ZRS1D - ZRDIFD*ORPARD) + (ZRS2D*ORPA
     1RD)*DCOS(PI*LD)) + ATOMAD*TEVD*(DCOS(TWOPI*(KD/3.0D0 +
     22.0D0*HD/3.0D0 + LD/4.0D0)) + DCOS(TWOPI*(2.0D0*KD/3.0
     33D0 + HD/3.0D0 + 0.75D0*LD)))
      STRFAC = STRF1C*STRF1C
      GO TO 4500
C
C     THIS IS THE ENTRY POINT WHEN ONLY ONE STRUCTURE FACTOR
C     IS CALC'D.
      ENTRY STRFAK (HD,KD,LD,THET2D,ATOMCD,ATOMAD,ORPARD,TEV
     1D,ZRS1D,ZRS2D)
      STRFAC = 0.0D0
 4500 CONTINUE
      STRF2C = ATOMCD*((ZRS1D - ZRDIFD*ORPARD) + (ZRS2D*ORPA
     1RD)*DCOS)PI*LD)) + ATOMAD*TEVD*(DCOS(TWOPI*(HD/3.0D0 +
     22.0D0*KD/3.0D0 + LD/4.0D0)) + DCOS(TWOPI*(2.0D0*HD/3.0
     33D0 + KD/3.0D0 + 0.75D0*LD)))
      STRFAC = STRFAC + STRF2C*STRF2C
      RETURN
      END
```

5. Calculation of Corrected D-Spacings and 2θ Values From Measured Lattice Parameters

This program calculates d-spacings and 2θ values from measured lattice parameters for specific planes in a hexagonal structure. A maximum of 50 sets of lattice parameters can be run at one time in this program.

```
C     THIS PROGRAM CALCULATES THE D-SPACINGS AND 2-THETA
C     VALUES OF SELECTED LINES FROM THE A AND C VALUES
C     OBTAINED BY INTERNAL STANDARD WORK.
C
      IMPLICIT REAL*8 (A-H,K-M,O-Z)
      DIMENSION A(50), C(50)
C
C     READ IN THE A AND C VALUES.
      N = 13
      READ (1,1100) (A(I), C(I), I = 1,N)
 1100 FORMAT (2D10.4)
      WRITE (3,1200) (A(I), C(I), I = 1,N)
 1200 FORMAT (/5X, 'THE VALUE FOR A IS', F8.4, 5X, 'THE VALU
     1E FOR C IS', F8.4)
C
C     LAMDA2 = LAMDA/2 FOR CU-KA RADIATION
      LAMDA2 = 0.77089D0
      TWOPI = 6.283185307179586
C
```

```
C       IF MORE THAN ONE SET OF A AND C VALUES ARE READ IN,
C       THEN THIS NEXT SECTION BECOMES A DO LOOP.
        DO 4000 I = 1,N
        A01 = 4.0D0/3.0D0*A(I)*A(I))
        A11 = 4.0D0/(A(I)*A(I))
        A02 = 16.0D0/(3.0D0*A(I)*A(I))
        A12 = 28.0D0/(3.0D0*A(I)*A(I))
        A03 = 12.0D0/(A(I)*A(I))
        C1 = 1.0D0/(C(I)*C(I))
        C2 = 4.0D0/(C(I)*C(I))
        C3 = 9.0D0/(C(I)*C(I))
        C4 = 16.0D0/(C(I)*C(I))
        C5 = 25.0D0/(C(I)*C(I))
        C6 = 36.0D0/(C(I)*C(I))
C
C       NOW FOR THE D-SPACING CALCULATIONS.  THE NUMBERS
C       FOLLOWING D ARE THE MILLER INDICES.
        D001 = C(I)
        D010 = 1.0D0/DSQRT(A01)
        D002 = C(I)/2.0D0
        D011 = 1.0D0/DSQRT(A01 + C1)
        D012 = 1.0D0/DSQRT(A01 +C2)
        D003 = C(I)/3.0D0
        D110 = 1.0D0/DSQRT(A11)
        D111 = 1.0D0/DSQRT(A11 + C1)
        D013 = 1.0D0/DSQRT(A01 + C3)
        D020 = 1.0D0/DSQRT(A02)
        D112 = 1.0D0/DSQRT(A11 + C2)
        D004 = C(I)/4.0D0
        D021 = 1.0D0/DSQRT(A02 + C1)
        D022 = 1.0D0/DSQRT(A01 + C2)
        D014 = 1.0D0/DSQRT(A01 + C4)
        D113 = 1.0D0/DSQRT(A11 + C3)
        D023 = 1.0D0/DSQRT(A02 + C3)
        D005 = C(I)/5.0D0
        D120 = 1.0D0/DSQRT(A12)
        D114 = 1.0D0/DSQRT(A11 + C4)
        D121 = 1.0D0/DSQRT(A12 + C1)
        D015 = 1.0D0/DSQRT(A01 + C5)
        D122 = 1.0D0/DSQRT(A12 + C2)
        D024 = 1.0D0/DSQRT(A02 + C4)
        D030 = 1.0D0/DSQRT(A03)
        D031 = 1.0D0/DSQRT(A03 + C1)
        D123 = 1.0D0/DSQRT(A12 + C3)
        D006 = C(I)/6.0D0
        D115 = 1.0D0/DSQRT(A11 + C5)
        D032 = 1.0D0/DSQRT(A03 + C2)
        D016 = 1.0D0/DSQRT(A01 + C6)
        D025 = 1.0D0/DSQRT(A02 + C5)
C
C       THIS SECTION CALCULATES THE 2-THETA VALUE FOR EACH
C       D-SPACING.  TT MEANS 2-THETA.  THE NUMBERS FOLLOWING
C       TT ARE THE MILLER INDICES.
```

```
C
      TT001 = DARSIN(LAMDA2/D001)*720.0D0/TWOPI
      TT010 = DARSIN(LAMDA2/D010)*720.0D0/TWOPI
      TT002 = DARSIN(LAMDA2/D002)*720.0D0/TWOPI
      TT011 = DARSIN(LAMDA2/D011)*720.0D0/TWOPI
      TT012 = DARSIN(LAMDA2/D012)*720.0D0/TWOPI
      TT003 = DARSIN(LAMDA2/D003)*720.0D0/TWOPI
      TT110 = DARSIN(LAMDA2/D110)*720.0D0/TWOPI
      TT111 = DARSIN(LAMDA2/D111)*720.0D0/TWOPI
      TT013 = DARSIN(LAMDA2/D013)*720.0D0/TWOPI
      TT020 = DARSIN(LAMDA2/D020)*720.0D0/TWOPI
      TT112 = DARSIN(LAMDA2/D112)*720.0D0/TWOPI
      TT004 = DARSIN(LAMDA2/D004)*720.0D0/TWOPI
      TT021 = DARSIN(LAMDA2/D021)*720.0D0/TWOPI
      TT022 = DARSIN(LAMDA2/D022)*720.0D0/TWOPI
      TT014 = DARSIN(LAMDA2/D014)*720.0D0/TWOPI
      TT113 = DARSIN(LAMDA2/D113)*720.0D0/TWOPI
      TT023 = DARSIN(LAMDA2/D023)*720.0D0/TWOPI
      TT005 = DARSIN(LAMDA2/D005)*720.0D0/TWOPI
      TT120 = DARSIN(LAMDA2/D120)*720.0D0/TWOPI
      TT114 = DARSIN(LAMDA2/D114)*720.0D0/TWOPI
      TT121 = DARSIN(LAMDA2/D121)*720.0D0/TWOPI
      TT015 = DARSIN(LAMDA2/D015)*720.0D0/TWOPI
      TT122 = DARSIN(LAMDA2/D122)*720.0D0/TWOPI
      TT024 = DARSIN(LAMDA2/D024)*720.0D0/TWOPI
      TT030 = DARSIN(LAMDA2/DC30)*720.0D0/TWOPI
      TT031 = DARSIN(LAMDA2/D031)*720.0D0/TWOPI
      TT123 = DARSIN(LAMDA2/D123)*720.0D0/TWOPI
      TT006 = DARSIN(LAMDA2/D006)*720.0D0/TWOPI
      TT115 = DARSIN(LAMDA2/D115)*720.0D0/TWOPI
      TT032 = DARSIN(LAMDA2/D032)*720.0D0/TWOPI
      TT016 = DARSIN(LAMDA2/D016)*720.0D0/TWOPI
      TT025 = DARSIN(LAMDA2/D025)*720.0D0/TWOPI
      WRITE (3,2000) A(I), C(I)
 2000 FORMAT (//1X, 'THE D-SPACINGS, AND 2-THETA VALUES BELO
     1W, FOR THE LATTICE PARAMETERS A=', F8.4, 3X, 'C=', F8.
     24, 3X, 'ARE:')
      WRITE (3,2100) D001, D010, D002, D011, D012, D003, D11
     10, D111, D013, D020, TT001, TT010, TT002, TT011, TT012
     2, TT003, TT110, TT111, TT013, TT020
 2100 FORMAT (/3X, 'D(001)', 4X, 'D(010)', 4X, 'D(002)', 4X,
     1'D(011)', 4X, 'D(012)', 4X, 'D(003)', 4X, 'D(110)', 4X
     2, 'D(111)', 4X, 'D)013)', 4X, 'D(020)'/3X, F6.4, 9(4X,
     3F6.4)/4X, F5.2, 9(5X, F5.2))
      WRITE (3,2101) D112, D004, D021, D022, D014, D113, D02
     13, D005, D120, D114, TT112, TT004, TT021, TT022, TT014
     2, TT113, TT023, TT005, TT120, TT114
 2101 FORMAT (/3X, 'D(112)', 4X, 'D(004)', 4X, 'D(021)', 4X,
     1'D(022)', 4X, 'D(014)', 4X, 'D(113)', 4X, 'D(023)', 4X
     2, 'D(005)', 4X, 'D(120)', 4X, 'D(114)'/3X, F6.4, 9(4X,
     3F6.4)/4X, F5.2, 9(5X, F5.2)
      WRITE (3,2102) D121, D015, D122, D024, D030, D031, D12
     13, D006, D115, D032, TT121, TT015, TT122, TT024, TT030
```

```
     2, TT031, TT123, TT006, TT115, TT032
2102 FORMAT (/3X, 'D(121)', 4X, 'D(015)', 4X, 'D(122)', 4X,
     1'D(024)', 4X, 'D(030)', 4X, 'D(031)', 4X, 'D(123)', 4X
     2, 'D(006)', 4X, 'D(115)', 4X, 'D(032)'/3X, F6.4, 9(4X,
     3F6.4)/4X, F5.2, 9(5X, F5.2))
      WRITE (3,2103) D016, D025, TT016, TT025
2103 FORMAT (/3X, 'D(016)', 4X, 'D(025)'/3X, F6.4, 4X, F6.4
     1/4X, F5.2, 5X, F5.2)
4000CONTINUE
      STOP
      END
```

APPENDIX B

X-RAY RESULTS FOR SAMPLES FIRED AT 817°C

Sample: 2.0-1 Analyzed composition: --

Sample color and texture: Golden brown powder sinter.

Sample tube color: Clear

Firing schedule: 817°C for 130 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.617	121	90	90
(?)	5.004	27	42	41
(002)	3.322	190	240	245
(?) +	3.087	44	66	66
(011)	3.038	100	100	100
(?)	2.690	71	78	71
(?)	2.498	36	43	39
(012) +	2.374	70	57	60
(?)	2.337	30	30	32
(003)	2.204	54	38	41
(110)	1.973	28	34	30
(013)	1.854	26	37	37
(112)	1.694	9	tl	11
(?) +	1.668	20	28	28
(004,021)	1.655	52	50	49
(?)	1.586	10	tl	15
(022)	1.519	10	tl	5
(014)	1.489	tl	tl	2
(113)	1.472	10	tl	8
(?)	1.430	tl	tl	13
(023)	1.351	tl	tl	1

Sample 2.0-1 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(005)	1.330	tl	tl	7
(120)	1.289	12	22	8
(114,121)	1.269	23	29	31
(?)	1.252	tl	15	8
(015)	1.236	15	--	--
(122)	1.204	tl	--	--
(024)	1.192	11	13	15

Sample: 2.0-2 Analyzed composition: --

Sample color and texture: Golden-brown powder sinter.

Sample tube color: Clear

Firing schedule: 817°C for 130 hours.

Comments: The relative intensity values are not too reliable, because there was noise in the scans, caused by excessive heat in the room.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.607	114	128	92
(?)	4.986	39	60	37
(002)	3.323	267	307	237
(?) +	3.084	48	47	47
(011)	3.037	100	100	100
(?)	2.693	42	60	48
(?)	2.497	43	45	45
(012) +	2.391	55	58	65
(?)	2.339	22	15	41
(003)	2.203	41	34	43
(110)	1.972	36	25	42
(013) +	1.854	24	24	16
(?)	1.815	28	17	16
(?)	1.773	18	17	12
(112)	1.691	--	7	--
(?) +	1.667	33	38	28
(004,021)	1.654	50	47	34
(?)	1.584	--	10	17
(022)	1.516	8	10	1
(014)	1.493	7	--	0
(113)	1.473	15	11	2

Sample 2.0-2 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(023)	1.351	14	--	--
(114,121)	1.268	33	27	16
(?)	1.250	14	--	0
(015)	1.234	19	15	0
(024)	1.192	12	12	13

Sample: 1.9-1 Analyzed composition: --

Sample color and texture: Brownish-black powder sinter plus a small amount of metallic material.

Sample tube color: Black

Firing schedule: 817°C for 130 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.677	41	28	27
(002)	3.336	44	33	29
(011)	3.056	100	100	100
(?)	2.898	3	3	4
(?)	2.546	4	tl	1
(?)	2.487	12	9	10
(012)	2.387	52	54	55
(003)	2.227	17	12	12
(110) + (?)	1.984 1.961	30	28	30
(013) + (?)	1.868 1.849	27	27	29
(112)	1.705	8	6	6
(004,021)	1.667	34	30	32
(022)	1.527	11	12	13
(014)	1.501	6	5	4
(113)	1.481	6	5	4
(023)	1.358	7	5	6
(114,121)	1.276	24	22	23
(015)	1.244	10	10	14
(122)	1.210	7	8	6
(030)	1.145	6	5	5
(123)	1.121	7	6	5
(016)	1.057	10	8	6

Sample: 1.9-2 Analyzed composition: --

Sample color and texture: Light black powder sinter.

Sample tube color: Black

Firing schedule: 817°C for 130 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.662	49	44	42
(?)	6.113	7	tl	1
(010) +	3.402	4	2	2
(002)	3.326	39	34	35
(011)	3.049	100	100	100
(012)	2.389	46	51	50
(003) +	2.217	13	12	14
(?)	2.195	1	--	--
(110)	1.980	29	26	27
(013)	1.863	22	21	24
(112)	1.703	6	6	6
(004,021)	1.662	32	34	32
(022)	1.530	10	12	13
(014)	1.497	2	3	2
(113)	1.478	4	6	1
(023)	1.358	5	5	3
(114,121)	1.274	22	24	23
(015)	1.243	7	7	7
(122)	1.203	8	9	7
(030)	1.143	4	--	--
(123)	1.120	4	--	--
(032)	1.083	tl	--	--
(016)	1.055	7	--	--

Sample: 1.8-1 Analyzed composition: --

Sample color and texture: Brownish-gray black powder sinter.

Sample tube color: Black

Firing schedule: 817°C for 130 hours.

Comments: The d-spacings for all lines below (021) have been taken from the count scan. The x-ray machine was noisy due to excessive heat in the room.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.622	46	34	34
(002)	3.318	38	30	31
(011)	3.045	100	100	100
(?)	2.643	3	--	1
(012)	2.386	50	46	44
(003)	2.215	22	12	13
(?)	2.096	8	--	0
(?)	2.049	8	--	2
(110)	1.979	30	30	29
(?)	1.898	7	--	0
(013)	1.862	21	18	17
(112)	1.702	7	6	5
(004,021)	1.661	30	28	26
(022)	1.659	--	9	8
(113)	1.476	--	5	2
(023)	1.355	--	7	5
(114,121)	1.272	--	22	21
(015)	1.240	--	6	6
(122)	1.207	--	8	7
(030)	1.143	--	tl	2
(123)	1.119	--	tl	5
(016)	1.054	--	tl	3

Sample: 1.8-2 Analyzed composition: --

Sample color and texture: Brown powder sinter.

Sample tube color: Brown smokey appearance.

Firing schedule: 817°C for 130 hours.

Comments: No count scan was run, because the x-ray machine was noisy due to excessive heat in the room. A second slow scan was run instead.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%(	Count Scan (Wt.%)	(Ct.%)
(001)	6.652	44	67	--
(002)	3.320	35	59	--
(011)	3.048	100	100	--
(?)	2.643	3	--	--
(012)	2.385	47	55	--
(003)	2.212	11	18	--
(110)	1.978	33	31	--
(013)	1.860	22	23	--
(112)	1.699	7	8	--
(004,021)	1.659	29	37	--
(022)	1.522	10	13	--
(014)	1.494	2	3	--
(113)	1.475	6	6	--
(023)	1.355	6	6	--
(114,121)	1.271	24	26	--
(015)	1.238	8	14	--
(122)	1.206	9	8	--
(030)	1.142	5	5	--
(123)	1.118	6	6	--
(025)	1.052	8	--	--

Sample: 1.7-1 Analyzed composition: --

Sample color and texture: Golden-brown powder sinter.

Sample tube color: Clear

Firing schedule: 817°C for 130 hours.

(001)	6.637	71	63	64
(?)	5.015	8	8	8
(002)	3.326	94	100	101
(?) +	3.099	12	14	14
(011)	3.045	100	100	100
(?)	2.700	14	17	16
(?)	2.506	9	11	9
(012)	2.383	46	47	44
(?)	2.345	7	4	4
(003)	2.210	23	27	24
(110)	1.977	23	26	23
(013)	1.858	28	24	23
(?)	1.821	--	6	3
(?)	1.783	--	6	5
(112)	1.699	6	5	3
(?) +	1.671	12	11	10
(004,021)	1.658	44	42	40
(022)	1.521	9	8	7
(014)	1.492	3	tl	1
(113)	1.474	6	7	8
(?)	1.432	--	tl	2
(023)	1.354	6	7	4
(114,121)	1.271	16	22	23
(015)	1.236	11	13	11
(122)	1.206	7	9	5
(024)	1.193	5	6	7
(123)	1.116	7	tl	5
(025)	1.050	11	10	12

Sample: 1.7-2 Analyzed composition: --

Sample color and texture: Grayish-brown powder sinter with some metallic material.

Sample tube color: Black

Firing schedule: 817°C for 130 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.642	42	17	17
(002)	3.324	35	18	21
(011)	3.044	100	100	100
(012)	2.387	51	50	42
(003)	2.216	9	8	16
(110)	1.978	25	31	28
(013)	1.863	22	20	17
(112)	1.701	9	5	5
(004,021)	1.662	22	23	24
(022)	1.523	9	8	9
(014)	1.497	5	2	0
(113)	1.475	6	--	--
(023)	1.356	9	8	10
(114,121)	1.274	28	24	24
(015)	1.243	12	--	7
(122)	1.209	9	--	6
(024)	1.180	2	--	--

Sample: 1.6-1 Analyzed composition:

Sample color and texture: Blackish-brown powder sinter with some gray metallic material.

Sample tube color: Black except for a clear portion at the top of the tube.

Firing schedule: 817°C for 130 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.717	30	23	20
(002)	3.344	29	19	17
(?)	3.242	18	29	24
(011) +	3.060	100	100	100
(?)	3.007	4	--	--
(?)	2.620	5	--	5
(?)	2.539	9	6	5
(?)	2.485	12	16	11
(012) +	2.396	63	54	51
(?)	2.346	6	6	3
(003)	2.226	12	10	10
(110)	1.994	41	38	34
(013) +	1.868	24	31	24
(?)	1.846	5	5	4
(112)	1.705	11	8	5
(004,021)	1.666	33	27	20
(022)	1.527	13	14	13
(014)	1.500	4	5	5
(113)	1.480	7	7	7
(?)	1.3800	6	tl	3
(023)	1.360	8	7	3
(114,121)	1.276	19	29	24
(015)	1.245	7	7	4

Sample 1.6-1 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(122)	1.211	9	12	7
(030)	1.145	6	9	6
(123)	1.122	8	5	7
(016)	1.058	9	11	8

Sample: 1.6-2 Analyzed composition: --

Sample color and texture: Golden-brown powder sinter.

Sample tube color: Clear

Firing schedule: 817°C for 130 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.652	64	63	63
(010) +	3.400	3	2	1
(002)	3.314	64	72	71
(011)	3.042	100	100	100
(?)	2.479	2	--	--
(012)	2.382	49	44	45
(003)	2.210	26	28	28
(110)	1.975	25	27	22
(111)	1.896	2	--	--
(013)	1.858	24	26	25
(112)	1.698	6	6	6
(004,021)	1.658	40	48	47
(022)	1.521	9	10	9
(014)	1.494	3	3	2
(113)	1.474	7	6	6
(023)	1.354	5	7	7
(114,121)	1.270	21	22	23
(015)	1.238	11	10	9
(122)	1.206	7	8	9
(030)	1.140	4	3	3
(123)	1.117	5	--	--
(032)	1.080	1	--	--
(025)	1.051	11	--	--

Sample: 1.5-2 Analyzed composition: --

Sample color and texture: Brownish-violet powder sinter plus some golden-brown metallic material.

Sample tube color: Clear

Firing schedule: 817°C for 130 hours.

Comments: No count scan was run, because the x-ray machine was noisy due to excessive heat in the room. A second slow scan was run instead.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.652	34	25	--
(002) +	3.323	38	35	--
(?)	3.239	92	93	--
(011)	3.043	100	100	--
(012) +	2.382	53	56	--
(?)	2.354	27	27	--
(003)	2.224	--	42	--
(112)	1.975	52	39	--
(013)	1.851	--	45	--
(112)	1.695	26	20	--
(004,021)	1.657	78	44	--
(022)	1.525	36	12	--
(121)	1.270	29	30	--
(015)	1.237	12	--	--
(025)	1.050	20	--	--

Sample: 1.5-3 Analyzed composition: --

Sample color and texture: Brown powder sinter plus light brown metallic core.

Sample tube color: Grayish-black and grayish brown-stripes around the tube.

Firing schedule: 817°C for 130 hours.

(001)	6.774	31	30	21
(002)	3.352	28	32	33
(011)	3.066	100	100	100
(012)	2.400	50	51	52
(003)	2.230	9	11	11
(110)	1.987	41	45	45
(013)	1.870	26	24	23
(112)	1.709	9	8	8
(004,021)	1.669	34	37	37
(022)	1.528	7	9	12
(014)	1.505	3	2	3
(113)	1.482	5	6	7
(023)	1.360	6	7	9
(114,121)	1.277	27	29	30
(015)	1.245	8	8	10
(122)	1.210	8	8	11
(030)	1.146	5	5	5
(123)	1.122	6	6	6
(025)	1.057	11	8	9

Sample: 1.4-1 Analyzed composition: --

Sample color and texture: Brown powder sinter.

Sample tube color: Clear

Firing schedule: 817°C for 130 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.369	2	2	1
(001)	6.662	81	81	81
(010) +	3.413	2	2	2
(002)	3.318	74	79	78
(?) +	3.108	1	2	2
(011)	3.044	100	100	100
(?)	2.698	2	2	2
(?)	2.638	2	1	1
(?) +	2.494	2	--	3
(?)	2.452	2	--	
(012) +	2.382	51	54	52
(?)	2.345	2	1	1
(003)	2.210	30	32	31
(110)	1.976	20	23	23
(013) +	1.856	28	28	27
(?)	1.830	2	2	2
(112)	1.698	8	6	5
(004,021)	1.655	49	48	48
(022)	1.520	11	9	9
(014)	1.491	3	4	3
(113)	1.473	6	7	6
(023)	1.350	6	6	6
(114,121)	1.269	19	24	23
(015)	1.236	11	16	15

Sample 1.4-1 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities		
		Slow Scan	Count Scan	
		(Wt.%)	(Wt.%)	(Ct.%)
(122)	1.205	7	8	7
(030)	1.140	4	3	3
(123)	1.116	4	5	5
(025)	1.050	12	13	13

Sample: 1.3-1 Analyzed composition: --

Sample color and texture: Grayish-brown powder sinter.

Sample tube color: Smokey coating.

Firing schedule: 817°C for 130 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.437	2	1	1
(001)	6.712	61	62	60
(?)	4.780	3	3	3
(010) +	3.431	1	2	2
(002)	3.330	56	60	60
(?) +	3.171	3	2	2
(011) +	3.055	100	100	100
(?)	2.929	2	2	1
(?)	2.640	2	2	1
(?)	2.476	2	1	2
(012)	2.390	54	62	59
(003)	2.216	21	21	21
(?)	2.064	1	1	1
(110)	1.982	24	29	28
(111) +	1.898	4	2	2
(013) +	1.862	29	30	27
(?)	1.839	1	1	1
(112)	1.701	9	8	8
(004,021)	1.661)	39	42	39
(022)	1.524	10	10	10
(014)	1.495	4	4	4
(113)	1.476	7	6	6
(023)	1.355	7	6	6
(114,121)	1.272	23	28	27

Sample 1.3-1 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(015)	1.238	11	12	11
(122)	1.207	8	9	8
(024)	1.192	1	1	1
(030)	1.143	4	4	4
(123)	1.118	5	6	5
(032)	1.079	4	2	1
(016)	1.052	11	11	11

Sample: 1.2-1 Analyzed composition: --

Sample color and texture: Black powder sinter.

Sample tube color: Black against the sample, gray above the sample.

Firing schedule: 817°C for 130 hours.

Comments: The diffraction lines in this x-ray pattern are much broader and are lower in intensity than corresponding lines of samples with higher Te/Zr mole ratio.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.687	3	--	--
(002) +	3.340	14	14	7
(?)	3.263	14	14	7
(011)	3.066	100	100	100
(?)	2.729	40	43	18
(?) +	2.491	37	39	15
(012)	2.419	34	34	13
(?)	2.269	3	tl	1
(003)	2.212	3	tl	1
(?)	2.111	11	10	4
(110) +	1.997	32	34	14
(?)	1.965	15	11	5
(?)	1.929	5	4	1
(013)	1.875	29	30	13
(112)	1.712	8	7	2
(004,021)	1.665	21	21	8
(?)	1.611	6	4	2
(022)	1.528	6	3	2
(014) +	1.501	9	10	4
(113)	1.484	3	2	1
(?)	1.366	8	5	2

Sample 1.2-1 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(023)	1.348	6	6	3
(114,121)	1.281	11	15	6
(?)	1.255	5	5	4
(024)	1.199	5	5	3
(?)	1.173	8	10	3
(?)	1.153	3	--	--
(031)	1.125	5	--	2
(032)	1.086	5	--	1
(016)	1.056	tl	--	--
(?)	1.039	2	--	0

Sample: 1.1-1 Analyzed composition: --

Sample color and texture: Blackish=brown powder sinter.

Sample tube color: Black

Firing schedule: 817°C for 130 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.728	22	22	13
(?)	5.464	13	12	12
(?)	3.632	16	15	--
(010)	3.418	10	14	8
(?)	3.271	32	94	87
(?) +	3.076	14	15	15
(011)	3.014	100	100	100
(?)	2.913	6	5	3
(?)	2.824	4	6	6
(?)	2.756	10	11	12
(?) +	2.687	14	20	51
(?) +	2.630	228	225	562
(?) +	2.545	70	76	191
(?) +	269	218	545	
(012) +	2.415	93	96	240
(?)	2.364	27	27	69
(?)	2.305	8	6	5
(003)	2.225	10	11	12
(?)	2.165	74	74	79
(?)	2.117	25	19	21
(?) +	1.962	8	5	19
(?) +	1.925	107	117	428
(111)? +	1.884	92	91	335
(013)	1.851	30	30	109

Sample 1.1-1

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	1.776	17	14	15
(020,112)	1.709	23	24	28
(004,021)	1.664	28	24	23
(?)	1.617	13	14	14
(022) +	1.532	61	48	50
($0$14)	1.503	81	72	78
(?)	1.421	13	11	10
(?)	1.381	37	37	42
(023)	1.348	36	23	26
(?)	1.333	17	10	12
(120)?	1.299	33	30	32
(114,121) +	1.274	10	9	10
(?) +	1.262	14	12	14
(?) +	1.248	15	13	15
(015)	1.235	21	20	23
(122) +	1.207	22	28	30
(024)	1.196	49	40	43
(?)	1.174	41	33	35
(030)	1.150	9	16	17
(032)	1.079	15	--	--
(025)	1.050	8	--	--
(?)	1.039	6	--	--

Sample: 1.0-1 Analyzed composition: --

Sample color and texture: Black powder sinter.

Sample tube color: Black

Firing schedule: 817°C for 130 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	4.606	8	5	14
(?)	3.635	6	4	4
(?)	3.545	6	--	1
(010)? +	3.381	8	9	10
(?)	3.288	128	123	128
(?) +	3.135	8	9	9
(011) +	3.070	100	100	100
(?)	3.010	10	9	8
(?) +	2.764	19	12	12
(?)	2.716	18	19	20
(?)	2.631	26	28	23
(?) +	2.549	4	--	--
(?) +	2.499	327	360	361
(?) +	2.452	59	54	55
(012)	2.405	10	9	8
(?)	2.294	5	--	0
(003)	2.222	4	--	0
(?)	2.166	4	--	8
(?) +	2.026	13	10	8
(?) +	1.964	32	31	26
(?)	1.934	53	52	45
(111)?	1.886	98	94	97
(?)	1.766	6	--	4
(020) +	1.712	2	--	--

Sample 1.0-1 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(112)	1.694	6	--	--
(004,021) +	1.663	44	45	49
(?)	1.632	15	18	19
(022) +	1.530	15	13	13
(014)	1.504	66	81	83
(?)	1.424	11	--	2
(023)	1.349	49	61	59
(?)	1.246	12	9	7
(015)	1.235	6	10	12
(024)	1.199	20	36	34
(?)	1.174	36	54	62
(032)	1.086	19	10	16
(?)	1.038	15	13	14

Sample: 0.9-1 Analyzed composition: --

Sample color and texture: Blackish-brown powder sinter with a black flakey coating.

Sample tube color: Black

Firing schedule: 817°C for 130 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.755	18	20	11
(?)	5.444	12	8	8
(?)	3.645	16	11	12
(010)	3.428	8	6	3
(?)	3.277	114	100	105
(011)	3.019	100	100	100
(?)	2.922	4	6	4
(?)	2.769	6	6	4
(?) +	2.695	8	9	9
(?) +	2.635	213	205	211
(?) +	2.550	57	52	54
(?) +	2.500	321	296	304
(012) +	2.418	80	78	80
(?)	2.367	31	32	33
(?) +	2.167	74	76	79
(?)	2.119	23	18	19
(110) +	1.976	14	--	--
(?) +	1.930	120	107	107
(111)? +	1.886	106	100	101
(013)	1.850	51	26	25
(?)	1.780	25	18	15
(020,112)	1.720	36	21	16
(004,021) +	1.666	50	27	29
(?)	1.632	25	12	12

Sample 0.9-1 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(022) +	1.533	63	56	57
(014)	1.504	105	96	97
(?)	1.423	22	16	16
(?)	1.382	45	36	38
(023) +	1.349	65	54	57
(?)	1.332	34	16	17
(120)?	1.301	47	25	29
(114,121)	1.274	21	8	4
(015)	1.248	18	--	--
(122) +	1.206	41	56	50
(024)	1.198	60	21	21
(?)	1.175	43	46	47

Sample: 0.8-1 Analyzed composition: --

Sample color and texture: Brownish-black powder sinter with a shiny black, flakey surface coating.

Sample tube color: Grayish-black

Firing schedule: 817°C for 130 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.762	21	21	16
(?)	5.451	11	11	11
(?)	3.643	16	13	14
(010)	3.428	10	8	7
(002) +	3.344	2	--	--
(?)	3.277	36	37	35
(011)	3.019	100	100	100
(?)	2.920	4	5	3
(?)	2.828	4	5	5
(?)	2.759	9	9	7
(?) +	2.697	20	20	19
(?) +	2.634	233	225	222
(?) +	2.548	74	69	68
(?) +	2.498	140	133	131
(012) +	2.417	88	82	81
(?)	2.366	32	35	34
(?)	2.310	7	5	5
(003)	2.228	13	7	8
(?) +	2.166	88	85	80
(?)	2.118	28	25	23
(?) +	1.926	95	84	84
(111) +	1.885	63	53	53
(013)	1.852	31	28	28
(?)	1.777	20	17	16

Sample 0.8-1 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(020) +	1.718	22	20	20
(112)	1.706			
(004,021)	1.664	12	10	10
(?)	1.630	4	5	1
(022) +	1.532	62	53	56
(014)	1.503	70	54	57
(?)	1.422	13	11	12
(?)	1.382	40	37	38
(023) +	1.348	18	15	16
(?)	1.333	22	18	19
(?)	1.299	32	31	33
(114,121)	1.274	7	5	7
(?)	1.262	5	4	4
(015)	1.236	13	7	7
(122) +	1.206	32	24	28
(024)	1.195	40	27	32
(?)	1.175	23	12	17
(030)	1.150	16	9	12
(123)	1.118	5	--	--
(115)	1.100	6	--	--
(032)	1.079	12	6	6
(025)	1.049	10	6	8

Sample: 0.75-1 Analyzed composition: --

Sample color and texture: Brownish-black powder sinter.

Sample tube color: Black

Firing schedule: 817°C for 130 hours.

Comments: This sample was furnace-cooled instead of air-quenched.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.657	18	19	18
(002) +	3.334	24	25	23
(?)	3.253	2	2	2
(?) +	3.139	1	3	3
(011)	3.056	100	100	100
(?) +	2.714	23	21	21
(?)	2.617	4	3	3
(?)	2.536	1	--	--
(?) +	2.485	10	8	8
(?) +	2.443	10	4	4
(012)	2.396	47	44	43
(003)	2.224	8	6	5
(?) +	2.154	1	2	1
(?)	2.108	6	6	4
(?) +	2.020	5	4	4
(110) +	1.987	32	34	29
(?)	1.958	5	5	4
(111)? +	1.917	5	4	3
(013)	1.869	27	27	22
(020,112)	1.707	9	8	6
(004,021) +	1.667	29	28	23
(?)	1.646	2	1	1

Sample 0.75-1 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	1.607	3	4	2
(022)	1.529	11	12	8
(014) +	1.499	4	4	3
(113)	1.482	4	4	3
(023)	1.361	7	7	4
(?) +	1.291	1	1	1
(114,121) +	1.278	24	26	20
(?) +	1.260	2	2	1
(?)	1.219	8	9	7

Sample: 0.67-1 Analyzed composition: --

Sample color and texture: Brownish-grey-black powder sinter

Sample tube color: Black with white tinges.

Firing schedule: 817°C for 130 hours.

Comments: This sample was furnace-cooled instead of air-quenched. This material was very hard to grind; the material was not soft. Free, or partially reacted, Zr was present in the weight gain sample. In addition to the peaks listed below, there was one broad range of peaks from about 74° to 78° 2θ that were unresolvable, the range of the (121) and (015) lines.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.635	37	30	28
(?)	5.379	13	14	15
(?)	3.620	16	12	--
(?)	3.405	14	14	11
(002)?	3.256	47	43	45
(?)	2.167	tl	--	--
(011)?	3.002	100	100	100
(?)	2.811	tl	tl	4
(?)	2.740	9	7	8
(?) +	2.676	26	19	20
(?) +	2.621	234	215	215
(?) +	2.537	98	89	90
(?) +	2.487	178	166	167
(?) +	2.452	25	23	23
(012) +	2.407	108	111	111
(?)	2.256	30	19	19
(?)	2.299	tl	tl	4
(003)	2.215	12	12	9

Sample 0.67-1 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?) +	2.158	95	80	76
(?)	2.110	38	31	29
(111)? +	1.919	91	75	68
(013) +	1.879	82	68	62
(?)	1.846	40	38	34
(?)	1.772	24	21	18
(020,112)	1.705	25	24	20
(004,021)	1.660	16	14	13
(?)	1.628	tl	--	--
(022)? +	1.529	71	74	62
(014)?	1.500	84	74	62
(?)	1.420	15	tl	9
(?)	1.379	45	42	35
(?) +	1.346	25	29	22
(?)	1.330	17	20	15
(120)?	1.297	39	34	36
(122) +	1.206	23	26	22
(024) +	1.194	65	51	44
(?)	1.173	40	35	30
(030)?	1.148	18	18	15
(123)	1.116	tl	tl	--

Sample: 0.50-1 Analyzed composition: --

Sample color and texture: Grey-black powder.

Sample tube color: Mottled-black in region of sample; a whitish-grey, almost metallic coating above sample.

Firing schedule: 817°C for 130 hours.

Comments: This sample was furnace-cooled instead of air-quenched. Free, or partially reacted, Zr was present in the weight gain sample. In addition to the lines listed below, there was a broad range of lines from 70.43° to 73.87° 2θ and from 79.00° to 82.56° 2θ. The (122) line was separable from this latter range.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.589	21	24	13
(?)	5.363	13	12	13
(?)	3.606	19	20	19
(010)?	3.396	10	9	5
(?)	3.216	tl	tl	1
(?)	3.142	14	19	19
(011)?	2.998	100	100	100
(?)	2.814	19	15	19
(?) +	2.741	67	68	71
(?) +	2.666	95	90	94
(?) +	2.619	259	225	234
(?) +	2.532	127	115	120
(?) +	2.468	168	168	175
(012) +	2.406	89	77	80
(?)	2.355	32	21	22
(?)	2.300	48	41	42
(003)?	2.216	71	65	62
(?)	2.158	81	63	72
(?)	2.105	39	40	37

Sample 0.50-1 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(110)?	1.994	11	10	7
(?)	1.969	10	9	7
(?) +	1.939	12	10	10
(?) +	1.917	109	113	111
(?) +	1.896	10	10	10
(013)? +	1.878	44	42	41
(?)	1.846	30	28	28
(?)	1.795	tl	8	8
(?)	1.769	31	30	30
(020)?	1.709	21	11	15
(?)	1.611	35	36	13
(?) +	1.561	23	11	7
(022) +	1.529	71	58	36
(014)	1.500	75	70	44
(?)	1.464	11	9	6
(?)	1.443	15	12	6
(?) +	1.418	14	8	32
(?) +	1.404	37	32	120
(?)	1.391	31	21	80
(?) +	1.378	39	32	119
(?) +	1.370	30	37	138
(?) +	1.362	16	--	--
(023)?	1.353	10	13	50
(122)?	1.204	44	39	21
(030)?	1.148	22	--	--
(032)?	1.084	27	--	--
(025)?	1.047	tl	--	--

APPENDIX C

X-RAY RESULTS OF SAMPLES FIRED AT 600°C

Sample: 2.0-3 Analyzed composition: --

Sample color and texture: Violet-brown powder sinter.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Comments: The d-spacings are from the count scan. Noise on the electrical line obliterated part of the pattern of the slow scan.

Line	d (uncorr.) (Å.)	Relative Intensities		
		Slow Scan	Count Scan	
		(Wt.%)	(Wt.%)	(Ct.%)
(001)	6.588	15	11	10
(010)	3.380	2	--	--
(002)	3.300	12	10	10
(011)	3.030	100	100	100
(012)	2.374	40	45	42
(003)	2.203	4	4	4
(110)	1.970	27	30	30
(111)	1.888	tl	tl	1
(013)	1.852	18	19	18
(112)	1.693	7	6	6
(004,021)	1.653	22	22	22
(022)	1.518	8	10	10
(113)	1.470	8	5	5
(023)	1.351	--	6	6
(114,121)	1.268	--	22	23
(015)	1.235	--	7	5
(122)	1.203	--	9	8
(030)	1.140	--	5	4

Sample 2.0-3 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities		
		Slow Scan	Count Scan	
		(Wt.%)	(Wt.%)	(Ct.%)
(123)	1.115	--	6	6
(025)	1.049	--	6	6

Sample: 1.9-3 Analyzed composition: --

Sample color and texture: Purplish-brown powder sinter.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Lattice parameters: a_o = 3.950, c_o = 6.626 Å.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (St.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.618	36	33	31
(010)	3.401	2	--	--
(002)	3.305	34	33	33
(?)	3.084	--	4	4
(011)	3.033	100	100	100
(?)	2.861	4	--	--
(?)	2.756	1	--	--
(?)	2.689	4	2	3
(?)	2.631	2	2	1
(?)	2.479	2	5	4
(012) +	2.375	42	43	41
(?)	2.332	2	3	2
(003)	2.202	10	13	11
(110)	1.972	25	32	30
(111)	1.889	2	2	2
(013) +	1.852	19	22	21
(?)	1.827	tl	--	--
(112)	1.693	6	6	6
(004,021)	1.654	26	28	26
(022)	1.518	10	8	9
(014)	1.489	2	--	--
(113)	1.471	6	7	6
(023)	1.351	5	6	6

Sample 1.9-3 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(114,121)	1.268	24	25	22
(015)	1.234	8	8	7
(122)	1.203	9	8	8
(030)	1.139	4	4	4
(123)	1.115	5	6	6
(032)	1.077	2	--	--
(025)	1.048	8	8	8

Sample: 1.8-3 Analyzed composition: --

Sample color and texture: Brown-black powder sinter.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Lattice parameters: a_o = 3.951, c_o = 6.618 Å.

Comment: There is no count scan, because the background was too noisy. A second slow scan was run.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	(Ct.%)
(001)	6.722	26	29	
(010)	3.410	2	1	
(002)	3.332	19	22	
(011)	3.055	100	100	
(?)	2.644	1	1	
(?) +	2.480	2	2	
(012)	2.389	44	48	
(003)	2.216	8	11	
(110)	1.981	26	28	
(111) +	1.895	2	2	
(013) +	1.861	19	23	
(?)	1.838	1	--	
(112)	1.701	6	8	
(004,021)	1.660	23	25	
(022)	1.523	8	9	
(014)	1.493	2	2	
(113)	1.476	4	5	
(023)	1.355	5	8	
(114,121)	1.271	23	24	
(015)	1.238	6	6	
(122)	1.206	8	9	

Sample 1.8-3 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	(Ct.%)
(024)	1.192	1	1	
(030)	1.142	4	4	
(123)	1.117	6	5	
(032)	1.079	1	1	
(025)	1.050	7	8	

Sample: 1.7-3s Analyzed composition: --

Sample color and texture: Brown and blackish-brown powder sinter.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Lattice parameters: a_o = 3.950, c_o = 6.628 Å.

Comments: Two-phase material. X-ray scan also run on metallic material.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.296	1	1	1
(001)	6.620	41	41	41
(?)	4.991	2	2	1
(010)	3.410	4	2	2
(002)	3.311	40	42	41
(?) +	3.104	3	2	2
(011)	3.037	100	100	100
(?)	2.918	1	--	--
(?)	2.694	5	4	4
(?)	2.632	1	1	1
(?) +	2.495	2	2	2
(?) +	2.452	2	--	--
(012) +	2.378	44	46	46
(?)	2.338	3	2	2
(003)	2.205	13	13	13
(?)	2.102	1	--	--
(?)	2.056	1	--	--
(110)	1.973	29	29	29
(111)	1.891	2	2	1
(013) +	1.854	22	19	19
(?)	1.824	2	2	2

Sample 1.7-3s (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	1.779	tl	tl	1
(112)	1.695	8	6	6
(004)? +	1.671	1	1	1
(021)	1.655	30	29	29
(?)	1.585	1	tl	1
(022)	1.519	10	9	9
(014) +	1.489	3	2	2
(113)	1.471	7	6	5
(?)	1.403	1	tl	0
(023)	1.351	7	6	5
(114,121)	1.268	23	24	23
(015)	1.235	9	10	9
(122)	1.204	10	8	8
(024)	1.190	1	tl	0
(030)	1.140	4	4	4
(123)	1.115	6	5	5
(032)	1.077	2	1	1
(016,025)	1.049	10	9	8

Sample: 1.7-3ℓ Analyzed composition: --

Sample color and texture: "Globs" of material, having a golden-brown metallic luster.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Comments: Two-phase material. X-ray scan also run on "solid" material.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.599	2	2	2
(001)	6.649	25	28	24
(010)	3.402	2	2	2
(002)	3.323	32	32	31
(?) +	3.135	5	2	2
(011) +	3.045	100	100	100
(?)	2.932	1	1	1
(?) +	2.698	9	9	9
(?) +	2.656	2	1	1
(?) +	2.622	7	8	8
(?) +	2.560	1	--	--
(?) +	2.535	8	7	6
(?) +	2.468	6	3	3
(?) +	2.441	8	11	10
(012) +	2.387	40	39	36
(?)	2.347	2	1	1
(?)	2.286	1	2	2
$(003)_a$ +	2.222	4	5	4
$(003)_b$ +	2.205	5	6	5
(?) +	2.160	2	3	2
(?) +	2.135	2	2	1
(?)	2.108	4	4	2

Sample 1.7-3ℓ (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?) +	2.008	3	3	2
(110) +	1.978	22	28	26
(?) +	1.948	5	5	4
(?)	1.930	4	3	3
(111) +	1.890	2	4	3
(013) +	1.869	11	14	12
(?)	1.851	13	14	13
(?)	1.767	tl	--	--
(020) +	1.725	2	1	1
(112) +	1.701	9	9	8
(004)? +	1.668	13	18	15
(021)	1.654	13	17	15
(?)	1.604	2	--	--
(022) +	1.522	11	3,6	
(?)	1.500	2	--	--
(014) +	1.488	2	--	--
(113)	1.472	3	2	2
(?)	1.394	1	2	1
(?) +	1.377	3	2	2
(?) +	1.362	2	4	4
(023)	1.353	2	1	1
(?) +	1.295	2	3	2
(114,121) +	1.278	15	15	12
(?) +	1.268	11	12	10
(?) +	1.258	4	3	2
(?) +	1.251	2	--	--
(?) +	1.244	4	5	4
(015)	1.235	6	5	4
(122) +	1.204	6	7	5

Sample 1.7-3ℓ (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(024)	1.193	1	--	--
(030) +		very broad		
(123)		" "		
(016,025)	1.049	6	7	6

Sample: 1.6-3 Analyzed composition: --

Sample color and texture: Brown powder sinter with some blackish-brown material and some golden-brown material.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Lattice parameters: a_o = 3.954, c_o = 6.635 Å.

Comments: X-ray machine became noisy; count scan finished as a slow scan.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.585	16	14	12
(010) +	3.390	2	2	2
(002)	3.306	14	14	15
(011) +	3.031	100	100	100
(?)	2.951	2	--	--
(?)	2.459	2	1	3
(012)	2.376	43	41	41
(003)	2.203	6	5	3
(110) +	1.972	30	29	32
(?)	1.947	1	--	--
(111) +	1.888	2	1	1
(013)	1.854	16	18	18
(112)	1.695	8	6	2
(004,021)	1.655	25	19	22
(022)	1.519	10	9	5
(014) +	1.489	2	2	--
(113)	1.471	5	5	--
(023)	1.352	6	5	--
(114,121)	1.269	23	19	--
(015)	1.236	5	5	--

Sample 1.6-3 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities		
		Slow Scan	Count Scan	
		(Wt.%)	(Wt.%)	(Ct.%)
(122)	1.204	10	7	--
(030)	1.140	4	4	--
(123)	1.116	6	5	--
(032)	1.075	2	2	--
(025)	1.050	7	6	--

Sample: 1.5-4s Analyzed composition: --

Sample color and texture: Brown powder sinter.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Lattice parameters: a_o = 3.952, c_o = 6.644 Å.

Comments: Two-phase material. X-ray scan also run on metallic material.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.634	18	16	16
(010) +	3.389	3	2	2
(002)	3.315	15	15	15
(011)	3.039	100	100	100
(?)	2.613	2	4	3
(?)	2.531	4	4	3
(?) +	2.448	4	4	4
(012)	2.382	48	46	44
(003)	2.211	6	5	6
(110)	1.975	30	30	31
(111) +	1.890	2	2	2
(013) +	1.858	18	17	17
(?)	1.836	1	1	1
(112)	1.697	3	6	5
(004,021)	1.657	27	20	19
(022)	1.521	11	10	8
(014) +	1.490	1	1	1
(113)	1.474	5	5	5
(023)	1.353	4	4	5
(114,121)	1.270	22	22	22
(015)	1.238	4	4	5

Sample 1.5-4s (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(122)	1.206	8	8	8
(030)	1.141	3	3	3
(123)	1.117	5	5	3
(032)	1.080	1	--	--
(025)	1.051	4	5	5

Sample 1.5-4ℓ Analyzed composition: --

Sample color and texture: "Globs having a light brown luster.

Firing schedule: 600°C for 130 hours.

Comments: Two-phase material. X-ray scan also run on "solid" phase.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.628	66	75	59
(001)	6.682	8	9	2
(?)	5.402	23	28	22
(010) +	3.401	24	25	31
(002)	3.335	14	11	14
(?) +	3.120	11	9	9
(011) +	3.050	100	100	100
(?)	3.003	41	50	50
(?)	2.802	6	4	6
(?)	2.746	8	5	4
(?) +	2.682	24	23	23
(?) +	2.621	117	113	113
(?) +	2.536	197	193	192
(?) +	2.469	20	26	25
(?) +	2.442	44	50	48
(012) +	2.406	223	226	216
(?)	2.355	17	14	13
(003)	2.215	10	10	12
(?) +	2.158	41	42	39
(?)	2.111	40	54	50
(110) +	1.982	19	19	8
(?)	1.954	9	22	10
(111) +	1.877	26	19	17

Sample 1.5-4ℓ (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(013)	1.846	86	73	64
(?)	1.769	16	14	21
(112) +	1.701	32	34	32
(004,021)	1.664	29	39	49
(?) +	1.538	10	14	--
(022) +	1.521	66	53	--
(014) +	1.495	44	45	--
(113)	1.477	8	--	--
(?)	1.378	37	38	--
(?)	1.297	44	--	--
(?)	1.251	21	--	--
(122)	1.200	34	--	--
(032)	1.075	12	--	--
(016)	1.055	8	--	--
(?)	1.045	12	--	--

Sample: 1.4-3 Analyzed composition: --

Sample color and texture: A powder sinter having a goldish-brown, black color.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Lattice parameters: a_o = 3.951, c_o = 6.631 Å.

Comments: Count scan was run 2 days after slow scan was run, because of goniometer problems. Count scan was finished as a slow scan because of excessive noise on the electrical line.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.662	18	14	12
(010) +	3.404	3	3	3
(002)	3.318	22	14	13
(011)	3.044	100	100	100
(?)	2.641	1	tl	1
(?) +	2.494	1	2	2
(?) +	2.464	9	12	13
(012)	2.383	48	42	46
(003)	2.211	6	5	6
(110)	1.977	35	34	62
(111) +	1.894	4	4	--
(013) +	1.858	20	21	--
(?)	1.831	1	--	--
(020,112) +	1.698	7	5	6
(004,021)	1.657	27	22	22
(022)	1.522	10	8	9
(014)	1.499	1	2	2
(113)	1.473	7	4	5
(?) +	1.368	3	4	--

Sample 1.4-3 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(023)	1.353	7	6	--
(114,121)	1.270	24	21	--
(015)	1.236	5	6	--
(122)	1.206	6	8	--
(030)	1.141	4	4	--
(123)	1.117	5	4	--
(032)	1.082	2	--	--
(025)	1.050	6	5	--

Sample: 1.3-3 Analyzed composition: --

Sample color and texture: Blackish-brown powder sinter.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Lattice parameters: a_o = 3.952, c_o = 6.635 Å.

Comments: Free, or partially reacted, Zr in weight gain sample.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.707	13	12	13
(010) +	3.406	2	2	3
(002)	3.334	11	10	12
(011) +	3.055	100	100	100
(?)	2.970	2	2	2
(?)	2.805	1	1	1
(?)	2.645	1	--	--
(?)	2.575	t1	--	--
(?) +	2.465	12	11	10
(012)	2.390	45	41	40
(003)	2.219	5	4	4
(110)	1.981	30	32	31
(111) +	1.897	6	6	5
(013)	1.862	17	17	15
(?)	1.797	1	--	--
(020,112)	1.702	6	6	5
(004,021)	1.661	23	20	19
(?)	1.619	--	2	1
(022)	1.526	12	8	8
(014)	1.495	2	2	2
(113) +	1.478	4	4	4

Sample 1.3-3 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	1.463	4	3	3
(?) +	1.370	3	3	3
(023)	1.355	8	7	8
(114,121)	1.272	22	20	20
(015)	1.239	4	4	5
(122)	1.207	8	7	8
(030)	1.143	5	3	3
(123)	1.118	5	5	5
(032)	1.034	3	2	2
(016,025)	1.054	13	5	6

Sample: 1.2-3 Analyzed composition: --

Sample color and texture: Brown powder sinter.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Lattice parameters: a_o = 3.952, c_o = 6.629 Å.

Comments: Last four lines are taken from the count scan because noise on the electrical line blotted out the lines on the slow scan.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.704	26	24	23
(010) +	3.416	2	2	2
(002)	3.331	23	22	21
(011)	3.054	100	100	100
(?)	2.645	2	1	1
(?) +	2.472	6	4	4
(012)	2.388	49	47	45
(003)	2.216	8	8	7
(110)	1.982	34	29	28
(111) +	1.899	3	2	2
(013)	1.861	22	21	19
(112)	1.701	8	5	5
(004,021)	1.660	24	22	21
(?)	1.622	3	1	1
(022)	1.523	9	8	9
(014) +	1.495	2	1	1
(113)	1.475	7	6	6
(?) +	1.369	1	1	1
(023)	1.355	6	6	5
(114,121)	1.271	24	23	22

Sample 1.2-3 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities		
		Slow Scan (Wt.%)	Count Scan (Wt.%)	(Ct.%)
(015)	1.238	8	6	6
(122) +	1.206	9	8	8
(024)?	1.197	1	--	--
(030)	1.142	5	4	4
(123)	1.118	5	5	5
(032)	1.080	2	--	1
(025)	1.051	7	6	6

Sample: 1.1-3 Analyzed composition: --

Sample color and texture: Blackish-brown powder sinter with some golden-brown material at bottom of tube.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Comments: Free, or partially reacted Zr present in weight gain sample. Weight gain data was not plotted, because sample was partially spilled by painters early in the run.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(011)	6.735	8	7	7
(010) +	3.413	2	3	3
(002)	3.350	9	8	9
(011) +	3.063	100	100	100
(?)	2.948	2	3	3
(?)	2.810	1	tl	1
(?)	2.544	1	--	--
(?) +	2.472	11	10	10
(012)	2.401	37	43	42
(003)	2.223	4	4	4
(110)	1.986	41	40	40
(111) +	1.896	5	5	5
(013)	1.869	15	15	15
(020,112) +	1.706	7	6	11
(004,021)	1.665	21	21	39
(?)	1.619	2	--	0
(022)	1.527	11	8	9
(014) +	1.496	2	1	2
(?) +	1.482	2	3	4
(113)	1.466	5	2	3
(?) +	1.370	--	6	6

Sample 1.1-3 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities		
		Slow Scan (Wt.%)	Count Scan (Wt.%)	(Ct.%)
(023)	1.356	--	5	4
(114,121)	1.274	22	21	22
(015)	1.241	--	2	3
(122)	1.210	7	7	6
(030)	1.145	--	2	3
(123)	1.120	--	2	4
(032)	1.085	--	2	2
(025)	1.053	--	3	3

Sample: 1.0-3 Analyzed composition: --

Sample color and texture: Blackish-brown powder sinter.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Comments: Free, or partially reacted, Zr present in weight gain sample.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.761	8	6	7
(010) +	3.436	1	1	1
(002)	3.361	12	8	8
(011)	3.069	100	100	100
(?)	2.810	2	3	3
(?)	2.583	2	1	2
(?) +	2.470	21	21	20
(012)	2.401	45	46	48
(003)	2.225	4	4	4
(110)	1.990	38	40	39
(111) +	1.900	5	6	6
(013)	1.871	16	14	16
(020,112) +	1.708	6	5	3
(004,021)	1.668	21	17	18
(?)	1.620	2	2	1
(022)	1.529	9	10	9
(?) +	1.490	4	1	1
(014) +	1.480		3	2
(113)	1.465	4	5	5
(?) +	1.370	8	7	8
(023)	1.356	5	5	5
(114,121)	1.277	21	22	21
(015)	1.244	3	2	2

Sample 1.0-3 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(122)	1.210	7	7	6
(123)	1.121	3	3	4
(032)	1.085	2	3	3
(016)	1.056	--	6	--
(?)	1.037	2	2	--

Sample: 0.9-3 Analyzed composition: --

Sample color and texture: Blackish-brown powder sinter

Sample tube color: Clear on bottom half where sample was, an opaque Te metal film above sample.

Firing schedule: 600°C for 130 hours.

Comments: A large amount of free, or partially reacted, Zr in weight gain sample.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.712	9	7	7
(010) +	3.407	3	1	1
(002)	3.345	9	8	10
(011) +	3.059	100	100	100
(?)	2.961	2	--	--
(?)	2.809	1	2	1
(?)	2.575	2	2	1
(?) +	2.592	2	--	--
(?) +	2.464	22	19	20
(012)	2.398	43	43	45
(003)	2.224	5	3	4
(110) +	1.987	35	34	35
(?)	1.958	2	4	4
(111) +	1.897	7	8	9
(013)	1.869	15	13	15
(020,112) +	1.705	6	3	4
(004,021)	1.667	21	17	18
(?)	1.618	2	3	3
(022)	1.528	10	7	8
(113)	1.464	4	6	6
(?) +	1.369	10	9	10
(023)	1.353	10	5	6

Sample 0.9-3 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities		
		Slow Scan (Wt.%)	Count Scan (Wt.%)	(Ct.%)
(114,121)	1.277	23	17	20
(122)	1.211	7	6	6
(032)	1.084	4	5	5

Sample: 0.8-3 Analyzed composition: --

Sample color and texture: Brown powder sinter.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Comments: Some free, or partially reacted, Zr present in weight gain sample.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.753	10	9	8
(?)	4.593	1	1	2
(010) +	3.428	13	2	2
(002)	3.350		11	10
(011)	3.064	100	100	100
(?)	2.818	8	72	74
(?)	2.653	1	1	1
(?)	2.582	2	2	2
(?) +	2.469	12	12	12
(012)	2.396	42	42	42
(003)	2.224	5	4	5
(110)	1.986	36	36	36
(111) +	1.901	4	5	5
(013)	1.867	16	17	18
(020,112) +	1.705	7	6	6
(004,021) +	1.664	24	22	23
(?)	1.623	1	--	--
(022) +	1.527	10	--	--
(014) +	1.498	2	--	--
(113) +	1.479	4	--	--
(?)	1.467	4	--	--
(?) +	1.371	4	5	5
(023)	1.357	6	6	6

Sample 0.8-3 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(114,121)	1.274	15	14	14
(015)	1.241	5	3	5
(122)	1.209	8	8	8
(030)	1.144	4	3	4
(123)	1.120	5	5	5
(032)	1.084	3	3	3
(016,025)	1.053	6	4	4

APPENDIX D

X-RAY RESULTS OF CHEMICALLY ANALYZED SAMPLES FIRED AT 817°C

Sample: 2.0-4 Analyzed composition: $ZrTe_{2.12}$

Sample color and texture: Golden-brown powder sinter with some free Te crystals.

Sample tube color: Clear

Firing schedule: 817°C for 130 hours.

Lattice parameters: $a_o = 3.951$, $c_o = 6.621$ Å.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
$(001)_a$ +	61735	12	15	15
$(001)_b$	6.588	44	41	40
$(002)_a$ +	3.350	5	4	3
$(002)_b$	3.301	35	36	34
(011)	3.032	100	100	100
(012)	2.374	44	49	45
(003)	2.203	12	13	14
(110)	1.971	29	30	27
(013)	1.852	25	23	25
(112)	1.693	8	tl	7
$(004,021)_a$ +	1.654	25	26	24
$(021)_b$	1.647	4	5	5
(022)	1.518	10	12	11
(014)	1.488	tl	--	--
(113)	1.470	tl	--	--
(023)	1.350	7	--	--
$(114,121)_a$ +	1.268	13	18	15
$(121)_b$	1.265	9	9	7
(015)	1.234	10	--	--

Sample 2.0-4 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(122)	1.203	8	--	--
(030)	1.138	tl	--	--
(123)	1.115	6	--	--
(016) + (025)	1.050 1.046	10	--	--

Sample: 1.8-4 Analyzed composition: $ZrTe_{1.91}$

Sample color and texture: Brown powder sinter

Sample tube color: Clear above sample, brown against sample.

Firing schedule: 817°C for 130 hours.

Lattice parameters: $a_o = 3.953$, $c_o = 6.633$ Å.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.260	5	tl	4
$(001)_a$ +	6.738	12	15	16
$(001)_b$	6.549	124	99	101
(010) +	3.400	2	2	2
$(002)_a$ +	3.348	6	4	4
$(002)_b$	3.292	93	83	86
(011)	3.023	100	100	100
(012)	2.370	51	51	52
(003)	2.200	32	28	31
(110)	1.969	28	29	28
(111)	1.887	tl	--	--
(013) +	1.850	23	24	23
(?)	1.828	1	2	2
(112)	1.692	7	6	8
$(004,021)_a$ +	1.653	42	45	44
$(021)_b$	1.646	5	7	7
(022)	1.516	11	10	9
(014)	1.489	tl	--	--
(113)	1.470	5	--	--
(023)	1.350	7	--	--
$(114,121)_a$ +	1.268	21	14	16
$(121)_b$	1.264	5	7	7

Sample 1.8-4 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
$(015)_a$ +	1.235	9	5	6
$(015)_b$	1.232	4	4	4
$(122)_a$ +	1.204	5	--	--
$(122)_b$	1.201	5	--	--
(030)	1.140	tl	--	--
(123)	1.115	6	--	--
(016) +	1.051	6	--	--
(025)	1.048	7	--	--

Sample: 1.9-6 Analyzed composition: $ZrTe_{1.86}$

Sample color and texture: Brown powder sinter with some free Te crystals.

Sample tube color: Opaque; dark black against sample, lighter black above sample.

Firing schedule: 817°C for 130 hours.

Lattice parameters: $a_o = 3.953$, $c_o = 6.633$ Å.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.257	6	tl	5
$(001)_a$ +	6.763	5	7	7
$(002)_b$	6.554	127	115	116
(?)	4.685	tl	--	--
(010) +	3.392	1	1	1
$(002)_a$ +	3.349	4	4	4
$(002)_b$	3.297	94	90	89
(011)	3.027	100	100	100
(012)	2.373	50	46	47
(003)	2.204	37	36	35
(110)	1.970	33	28	29
(013) +	1.853	26	25	16
(?)	1.835	2	2	1
(112)	1.694	7	7	1
$(004,021)_a$ +	1.655	46	61	52
$(021)_b$	1.648	8	9	7
(022)	1.518	12	11	13
(014)	1.490	6	--	--
(113)	1.471	10	6	4
(023)	1.352	8	5	4
$(114,121)_a$ +	1.269	22	20	24

Sample 1.9-6 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
$(121)_b$	1.266	8	9	11
$(015)_a$ +	1.237	8	9	7
$(015)_b$	1.233	4	3	2
(122)	1.204	13	10	10
(030)	1.140	6	--	--
(123)	1.116	6	--	--
(016) +	1.052	6	--	--
(025)	1.049	5	--	--

Sample: 1.7-4s
1.7-4ℓ

Analyzed composition: $ZrTe_{1.65}$
$ZrTe_{1.47}$

Sample color and texture: Brown powder sinter with a hard metallic center core.

Sample tube color: Opaque; brown against samle, blackish-gray at top due to free Te crystals.

Firing schedule: 817°C for 130 hours.

Lattice parameters: $a_o = 3.956$, $c_o = 6.636$ Å.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	9.309	4	√	--
(?)	7.296	tl	√	--
$(001)_a$ +	6.748	5	5	5
$(001)_b$	6.573	67	83	83
(?)	4.708	6	5	4
(?)	3.649	tl	--	--
(010) +	3.406	2	2	2
$(002)_a$ +	3.351	5	7	7
$(002)_b$	3.302	49	53	55
(?)	3.144	tl	--	--
(011)	3.029	100	100	100
(?)	2.903	tl	--	--
(?)	2.441	tl	--	--
$(012)_a$ +	2.396	2	3	3
$(012)_b$	2.375	41	49	47
(?)	2.269	tl	--	--
$(003)_a$ +	2.220	3	--	--
$(003)_b$	2.206	18	20	20
(?) +	1.987	3	8	8
(110)	1.971	22	27	26
(111)	1.890	tl	--	--

Sample 1.7-4 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?) +	1.868	4	tl	--
(013)	1.854	22	26	23
(020) +	1.706	3	--	--
(112)	1.694	7	8	7
(?) +	1.667	5	6	5
(004,021)	1.655	36	38	33
(?)	1.557	tl	--	--
(022)	1.519	10	11	11
(014)	1.493	tl	--	--
(113)	1.472	5	--	--
(023)	1.351	9	8	9
(?) +	1.276	4	6	6
$(114,121)_a$ +	1.270	15	18	18
$(121)_b$	1.266	6	5	5
(015)	1.236	9	7	11
$(122)_a$ +	1.205	5	8	--
$(122)_b$	1.202	4	3	--
(030)	1.140	tl	--	--
$(123)_a$ +	1.117	tl	--	--
$(123)_b$	1.114	tl	--	--
(016) +	1.053	tl	--	--
(025)	1.049	tl	--	--

Sample: 1.6-6s Analyzed composition: $ZrTe_{1.69}$
1.6-6ℓ $ZrTe_{1.30}$

Sample color and texture: Brown powder sinter plus light brown, hard, metallic material.

Sample tube color: Opaque above sample, brown against sample.

Firing schedule: 817°C for 130 hours.

Lattice parameters: $a_o = 3.958$, $c_o = 6.644$ Å.

Comments: The (016) and (025) peaks were unresolvable.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
$(001)_a$ +	6.823	8	7	6
$(001)_b$	6.632	54	52	49
(?)	4.730	6	7	6
$(002)_a$ +	3.367	5	✓	✓
$(002)_b$	3.318	41	42	39
(?)	3.251	4	6	5
(011)	3.042	100	100	100
(?) +	2.644	4	--	--
(?)	2.612	4	--	--
(?) +	2.530	7	5	4
(?) +	2.484	19	21	18
(?) +	2.438	7	--	--
$(012)_a$ +	2.406	6	11	10
$(012)_b$	2.384	46	46	40
(003)	2.213	15	13	15
(110)	1.976	27	25	24
(111) +	1.895	4	4	3
(?) +	1.877	6	7	5
(013) +	1.860	22	25	19
(?)	1.839	5	3	2

Sample 1.6-6 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(112)	1.700	9	tl	6
(?) +	1.672	5	--	--
(004,021)	1.659	37	28	28
(022)	1.522	12	8	9
(014)	1.497	8	6	6
(113)	1.477	6	--	--
(023)	1.355	8	--	--
(?) +	1.279	4	tl	--
$(114,121)_a$ +	1.272	16	16	15
$(121)_b$	1.268	5	tl	--
(015)	1.239	9	tl	8
(122)	1.205	6	tl	7
(?)	1.172	tl	--	--
(030)	1.142	5	--	--
(123)	1.118	6	--	--
(016) + (p25)	--	12	--	--

Sample: 1.5-5s
1.5-5ℓ

Analyzed composition: -- $ZrTe_{1.39}$

Sample color and texture: Brown powder sinter plus golden-brown, hard, metallic material.

Sample tube color: Opaque, brownish color.

Firing schedule: 817°C for 130 hours.

Lattice parameters: a_o = 3.957, c_o = 6.643 Å.

Comments: The 1.5-5s chemical analysis sample was spilled.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.323	tl	--	--
$(001)_a$ +	6.794	5	7	6
$(001)_b$	6.602	102	91	80
(?)	4.720	7	tl	6
(010) +	3.415	2	tl	--
$(002)_a$ +	3.359	6	6	6
$(002)_b$ +	3.308	76	71	72
(?)	3.246	2	tl	--
(?)	3.151	5	tl	4
(011)	3.035	100	100	100
(?) +	2.482	7	8	8
(?)	2.445	3	2	2
$(012)_a$ +	2.403	4	5	4
$(012)_b$	2.379	51	52	48
(003)	2.209	28	28	26
(?) +	1.984	5	tl	--
(110) +	1.972	22	28	25
(?)	1.952	3	tl	--
(111) +	1.892	3	tl	--
(?) +	1.874	4	tl	--

Sample 1.5-5 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(013) +	1.856	26	29	31
(?)	1.836	4	tl	--
(112)	1.695	7	tl	7
(?) +	1.669	7	5	5
(004,021)	1.657	50	54	47
(022)	1.520	9	10	12
(014)	1.493	tl	--	--
(113)	1.473	5	--	5
(023)	1.353	8	--	8
(?) +	1.277	3	--	--
$(114,121)_a$ +	1.270	17	--	--
$(121)_b$	1.269	5	--	--
(015)	1.237	12	--	12
$(122)_a$ +	1.206	6	--	--
$(122)_b$	1.203	2	--	--
(024)	1.192	tl	--	--
(030)	1.140	tl	--	--
(123)	1.117	tl	--	--
(016) +	1.054	tl	--	--
(025)	1.050			

Sample: 1.4-2 Analyzed composition: $ZrTe_{1.52}$

Sample color and texture: Medium black powder sinter.

Sample tube color: Black

Firing schedule: 817°C for 130 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	9.349	tl	--	--
(001)	6.697	8	11	12
(?)	4.585	tl	--	--
(?)	3.591	tl	--	--
(010) +	3.397	4	3	2
(002)	3.344	26	26	25
(011)	3.068	100	100	100
(?) +	2.726	40	33	36
(?)	2.668	6	5	5
(?)	2.473	tl	tl	2
(012)	2.402	35	34	31
(003)	2.228	9	8	8
(?)	2.112	11	8	9
(110)	1.992	47	46	44
(?)	1.908	tl	--	--
(013)	1.871	27	24	24
(112)	1.710	7	8	7
(004,021)	1.670	32	29	28
(?)	1.607	8	8	6
(022)	1.533	7	6	5
(014)	1.503	tl	tl	1
(113)	1.484	6	5	4
(?)	1.448	3	3	3
(023)	1.364	9	8	7

Sample 1.4-2 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(114,121)	1.280	30	30	29
(015)	1.249	12	9	10
(122)	1.215	6	6	4
(?)	1.170	3	--	--
(030)	1.149	10	8	8
(123)	1.125	6	7	6
(032)	1.089	tl	--	--
(016)	1.059	5	--	--

Sample: 1.0-2 Analyzed Composition: $ZrTe_{1.11}$

Sample color and texture: Black powder sinter.

Sample tube color: Black, mottled against sample.

Firing schedule: 817°C for 130 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(002) +	3.309	9	12	13
(?)	3.240	25	22	24
(011)	3.048	100	100	100
(?)	2.713	40	43	40
(?) +	2.479	48	51	42
(012)	2.412	40	40	33
(?)	2.104	13	9	8
(110) +	1.992	36	32	29
(?)	1.956	18	17	16
(?)	1.922	7	6	4
(013)	1.871	30	29	23
(112)	1.709	tl	--	--
(004,021)	1.660	23	24	18
(?)	1.606	tl	--	--
(022)	1.527	tl	--	--
(014)	1.497	10	11	8
(?)	1.360	tl	--	--
(023)	1.346	8	--	--
(114,121)	1.278	10	--	--
(024)	1.195	tl	--	--
(?)	1.172	12	16	12

Sample: 1.1-2 Analyzed composition: $ZrTe_{1.05}$

Sample color and texture: Brownish-black powder sinter.

Sample tube color: Mottled white.

Firing schedule: 817°C for 130 hours.

Comments: There was a broad band of unresolvable lines from 42.50° to 44.15° 2θ.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(010) +	3.385	2	--	--
(002) +	3.324	9	--	--
(?) +	3.248	98	98	98
(?) +	3.150	38	44	44
(011) +	3.052	100	100	100
(?)	2.981	11	12	12
(?)	2.834	14	14	10
(?) +	2.752	17	10	11
(?) +	2.710	50	44	47
(?) +	2.617	32	26	27
(?) +	2.553	23	15	16
(?) +	2.438	285	281	302
(?) +	2.436	31	32	34
(012)	2.398	10	10	11
(003)	2.210	7	--	--
(?) +	2.018	21	21	22
(110) +	1.997	16	12	13
(?) +	1.963	31	22	24
(?) +	1.925	44	36	38
(013) +	1.877	104	90	95
(?)	1.848	18	13	14
(?)	1.810	tl	--	--

Sample 1.1-2 (cont.)

Line	d (uncorr.) Å.	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(112) +	1.693	16	--	--
(004,021) +	1.657	58	49	61
(?) +	1.626	17	15	18
(?) +	1.608	tl	--	--
(?)	1.594	tl	--	--
(022) +	1.532	18	tl	--
(014) +	1.498	78	74	--
(113)	1.481	6	tl	--
(?) +	1.358	3	5	4
(023)?	1.345	51	55	48
(?)	1.324	tl	--	--
(?)	1.260	tl	--	--
(015) +	1.243	13	--	--
(?)	1.230	11	--	--
(024)	1.196	30	29	26
(?)	1.172	63	60	57

Sample: 0.9-2 Analyzed composition: $ZrTe_{0.92}$

Sample color and texture: Light brownish-black powder sinter.

Sample tube color: Blackish-gray

Firing schedule: 817°C for 130 hours.

Comments: The most intense line is not the (011). There is a broad band of unresolvable lines from 75.95° to 78.00° 2θ.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.576	21	17	10
(?)	5.343	12	tl	8
(?)	3.604	19	18	20
(?)	3.494	tl	--	--
(010)	3.394	10	--	--
(?) +	3.242	224	180	181
(?)	3.149	16	18	18
(011) +	3.036	17	14	14
(?)	2.991	100	100	100
(?)	2.899	tl	--	--
(?)	2.824	tl	--	--
(?)	2.755	tl	--	--
(?) +	2.645	13	8	8
(?) +	2.614	233	225	230
(?) +	2.534	64	60	62
(?) +	2.481	346	313	321
(012) +	2.401	89	81	83
(?)	2.349	68	56	57
(003)	2.224	26	21	20
(?) +	2.154	91	85	85
(?) +	2.106	31	25	25

Sample 0.9-2 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	2.077	12	7	7
(?) +	1.915	124	116	109
(013) +	1.876	124	105	98
(?)	1.841	41	38	36
(?)	1.769	25	20	20
(112)	1.703	28	27	26
(004,021)	1.656	40	33	30
(?)	1.623	21	tl	10
(?)	1.559	tl	--	--
(?) +	1.532	47	22	19
(022) +	1.520	34	65	56
(014) +	1.498	114	111	97
(113)	1.477	10	10	9
(?)	1.417	23	27	24
(?)	1.377	56	59	52
(023)? +	1.361	64	57	47
(005)	1.329	24	28	22
(120)	1.296	32	31	31
(114,121)	1.271	15	--	--
(?)	1.259	16	--	--
(122) +	1.203	44	41	35
(024)	1.193	56	59	50
(?)	1.172	76	61	55
(030)	1.148	16	--	--
(123)	1.115	tl	--	--
(?)	1.098	tl	--	--
(?) +	1.084	13	--	--
(032)	1.077	22	--	--
(025)	1.047	tl	--	--
(?)	1.037	tl	--	--

Sample: 1.2-2 Analyzed composition: $ZrTe_{0.82}$

Sample color and texture: Blackish=brown powder sinter.

Sample tube color: Black

Firing schedule: 817°C for 130 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.576	10	--	--
(?)	5.369	tl	--	--
(?)	4.538	tl	--	--
(?)	3.598	12	9	9
(010)? +	3.382	tl	8	7
(002)? +	3.325	tl	11	10
(?) +	3.244	155	170	170
(?) +	3.193	10	11	10
(?) +	3.117	16	16	16
(011) +	3.047	100	100	100
(?)	2.994	34	37	37
(?) +	2.746	18	12	11
(?) +	2.702	32	29	28
(?)	2.616	81	82	78
(?) +	2.533	24	14	14
(?) +	2.482	312	304	305
(?) +	2.435	50	44	44
(012) +	2.401	34	30	30
(?)	2.348	17	21	21
(003)	2.221	11	14	11
(?) +	2.155	28	34	34
(?) +	2.108	22	19	19
(?)	2.071	8	6	6
(?) +	2.018	17	18	17

Sample 1.2-2 (cont.)

Line	d (uncorr.) Å.	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(110) +	1.995	8	--	--
(?) +	1.960	36	40	36
(?) +	1.922	75	83	76
(013) +	1.877	117	110	101
(?)	1.840	15	27	24
(?)	1.767	13	10	12
(112) +	1.704	24	24	23
(004,021) +	1.657	57	45	44
(?)	1.622	33	28	28
(?) +	1.536	33	35	34
(022) +	1.524			
(014) +	1.498	78	88	87
(113)	1.477	10	10	10
(?)	1.419	tl	--	--
(?)	1.379	16	23	24
(023)? +	1.345	50	60	58
(005)?	1.331	8	7	7
(?) +	1.306	8	7	7
(120)?	1.295	15	19	17
(?) +	1.242	11	--	--
(015)	1.232	13	--	--
(122) +	1.204	13	18	18
(024)	1.195	37	40	39
(?)	1.172	53	54	54
(030)	1.148	tl	--	--
(032)	1.084	11	--	--
(?)	1.037	12	--	--

Sample: 1.3-2 Analyzed composition: $ZrTe_{0.78}$

Sample color and texture: Brownish-black powder sinter.

Sample tube color: Grayish-black

Firing schedule: 817°C for 130 hours.

Comments: The analyzed composition may be in error because holes were beginning to appear in the Pt crucible. The presence of many extra lines indicates, however, that the sample has a low Te/Zr mole ratio. The (030) and (123) lines are one low broad band.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.602	10	11	6
(?)	5.366	10	tl	9
(?)	4.557	17	12	12
(?)	3.613	8	--	--
(010) +	3.390	10	10	9
(002) +	3.323	8	14	12
(?)	3.251	73	77	69
(?) +	3.116	9	13	13
(011) +	3.054	100	100	100
(?)	2.997	61	57	57
(?)	2.896	tl	--	--
(?) +	2.806	5	--	--
(?) +	2.714	66	61	58
(?) +	2.617	128	141	133
(?)	2.533	37	45	43
(?) +	2.485	200	232	220
(?) +	2.438	34	44	41
(012) +	2.403	62	65	62
(?)	2.354	15	14	13
(003)	2.214	7	--	--

Sample 1.3-2 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?) +	2.156	45	51	46
(?)	2.106	25	27	24
(?) +	2.016	17	16	14
(110) +	1.989	9	10	9
(?) +	1.958	27	31	27
(?) +	1.920	62	70	63
(013)? +	1.877	90	94	84
(?)	1.843	18	20	18
(?)	1.769	10	7	8
(?) +	1.730	--	5	4
(112) +	1.704	31	26	20
(021)? +	1.658	50	51	40
(?) +	1.626	16	15	12
(?)	1.602	8	12	9
(?) +	1.535	18	15	14
(022) +	1.522	27	31	28
(014) +	1.498	73	77	70
(113)	1.477	9	4	4
(?)	1.418	tl	--	--
(?) +	1.378	27	26	23
(023)? +	1.360	10	6	5
(?) +	1.345	37	40	35
(?)	1.329	12	10	9
(120) +	1.296	32	--	--
(114,121)? +	1.275	15	--	--
(?) +	1.260	18	--	--
(015)? +	1.245	17	--	--
(?)	1.231	11	--	--
(122) +	1.204	16	10	10

Sample 1.3-2 (cont.)

Line	d (uncorr.) Å.	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(024)	1.194	42	35	33
(?)	1.172	42	37	35
(032) +	1.085	12	--	--
(?)	1.078	10	--	--
(025)? +	1.048	11	--	--
(?)	1.038	16	--	--

Sample: 0.8-2 Analyzed composition: $ZrTe_{0.72}$

Sample color and texture: Grayish-brown black powder sinter.

Sample tube color: Blackish-gray

Firing schedule: 817°C for 130 hours.

Comments: The most intense line is not the (011). The count scan was not completed due to problems with the airconditioner.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.628	30	20	10
(?)	5.379	tl	tl	--
(?)	4.606	tl	tl	9
(?)	3.603	15	12	13
(010) +	3.384	tl	7	8
(002) +	3.319	tl	--	--
(?) +	3.250	166	162	184
(?)	3.206	38	26	29
(011) +	3.059	29	29	29
(?)	2.997	100	100	100
(?)	2.896	tl	--	--
(?) +	2.738	24	14	--
(?) +	2.688	18	16	--
(?) +	2.617	239	208	--
(?) +	2.534	62	--	--
(?) +	2.483	347	--	--
(012) +	2.403	89	--	--
(?)	2.351	60	--	--
(003)	2.223	23	--	--
(?) +	2.156	93	--	--
(?) +	2.108	39	--	--

Sample 0.8-2 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	2.073	11	--	--
(?) +	1.919	113	--	--
(013) +	1.877	121	--	--
(?)	1.843	46	--	--
(?)	1.770	27	--	--
(112)	1.703	36	--	--
(004,021)	1.658	54	--	--
(?)	1.623	40	--	--
(022)	1.527	62	--	--
(014)	1.499	116	--	--
(113)	1.475	tl	--	--
(?)	1.418	25	--	--
(?)	1.378	51	--	--
(023) +	1.345	65	--	--
(005)	1.329	24	--	--
(120)	1.296	35	--	--
(122) +	1.204	35	--	--
(024)	1.194	70	--	--
(?)	1.172	87	--	--
(030)	1.147	tl	--	--

APPENDIX E

X-RAY RESULTS OF CHEMICALLY ANALYZED SAMPLES FIRED AT 600°C

Sample: 2.0-6 Analyzed composition: $ZrTe_{2.02}$

Sample color and texture: Brown powder sinter.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Lattice parameters: $a_o = 3.948$, $c_o = 6.622$ Å.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	8.805	tl	--	--
(?)	7.329	tl	--	--
$(001)_a$ + $(001)_b$	6.776 6.602	58	50	54
$(002)_a$ +	3.371	5	5	5
$(002)_b$	3.308	41	40	41
(?) +	3.096	4	3	3
(011)	3.037	100	100	100
(012)	2.378	46	44	46
(003)	2.205	14	13	14
(110)	1.974	27	29	25
(111)	1.892	tl	--	--
(013)	1.854	21	22	22
(112)	1.697	8	6	9
(004,021)	1.655	31	30	32
(022)	1.519	10	10	9
(014)	1.490	tl	--	--
(113)	1.472	6	7	4

Sample 2.0-6 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(023)	1.352	6	5	12*
(114,121)	1.268	26	24	28
(015)	1.234	9	8	9
(122)	1.204	9	8	9
(030)	1.139	tl	--	--
(123)	1.115	7	--	--
(025)	1.049	11	--	--

*high due to noise on the electrical line

Sample: 1.9-4 Analyzed composition: $ZrTe_{1.70}$

Sample color and texture: Brown powder sinter

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Lattice parameters: a_o = 3.952, c_o = 6.632 Å.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.260	tl	--	--
$(001)_a$ + $(001)_b$	6.697 6.520	42	41	38
(010) +	3.380	2	2	2
$(002)_a$ + $(002)_b$	3.337 3.284	4 26	4 25	3 24
(011)	3.015	100	100	100
(012)	2.365	43	46	44
(003)	2.197	10	10	10
(110)	1.966	26	30	27
(111)	1.885	tl	--	--
(?) +	1.864	tl	2	3
(013) +	1.848	23	24	25
(?)	1.828	tl	--	--
(112)	1.690	6	8	8
(004,021)	1.650	26	28	20
(022)	1.515	10	12	6
(014)	1.485	tl	--	--
(113)	1.468	5	7	5
(?)	1.365	tl	--	--
(023)	1.349	5	7	6
(114,121)	1.266	22	23	23
(015)	1.233	9	7	7

Sample 1.9-4 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(122)	1.202	8	7	8
(024)	1.188	tl	--	--
(030)	1.139	5	--	--
(123)	1.114	6	--	--
(025)	1.048	9	--	--

Sample: 1.6-4 Analyzed composition: $ZrTe_{1.67}$

Sample color and texture: Dark brown powder sinter.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Lattice parameters: a_o = 3.958, c_o = 6.642 Å.

Comments: A few metallic particles were present in the chemical analysis sample.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
$(001)_a$ +	6.722	29	24	29
$(001)_b$	6.559			
(010) +	3.394	2	1	1
$(002)_a$ +	3.348	4	4	4
$(002)_b$	3.297	13	14	13
(011)	3.024	100	100	100
(?) +	2.476	tl	--	--
(?)	2.450	tl	--	--
(012)	2.372	42	42	42
(003) +	2.203	6	5	6
(?)	2.180	1	1	1
(110)	1.970	34	33	32
(111)	1.884	tl	--	--
(013) +	1.852	17	17	17
(?)	1.833	1	--	--
(112)	1.694	7	7	7
(004,021)	1.653	23	25	22
(022)	1.518	11	12	10
(014)	1.489	tl	--	--
(113)	1.470	6	4	6
(023)	1.351	7	6	6

Sample 1.6-4 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
$(114,121)_a$ + $(121)_b$	1.269 1.264	25	24	23
(015)	1.236	6	5	7
(122)	1.204	9	9	9
(030)	1.130	6	--	--
(123)	1.115	6	--	--
(025)	1.050	7	--	--

Sample: 1.5-1s Analyzed composition: $ZrTe_{1.62}$
1.5-1ℓ $ZrTe_{1.53}$

Sample color and texture: Brown powder sinter plus golden-brown hard globules.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Lattice parameters: a_o = 3.952, c_o = 6.621 Å.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
$(001)_a$ + $(001)_b$	6.739 6.583	50	48	58
(010) +	3.404	2	--	--
$(002)_a$ +	3.353	6	8	9
$(002)_b$	3.302	27	26	29
(?)	3.111	tl	--	--
(011)	3.032	100	100	100
(?)	2.626	tl	--	--
(?)	2.532	51	--	--
(?) +	2.399	5	4	4
(012)	2.375	41	40	43
(003)	2.203	10	9	9
(110)	1.972	27	27	30
(111) +	1.886	2	--	--
(?) +	1.867	4	--	--
(013)	1.852	20	18	18
(020) +	1.709	2	1	1
(112)	1.694	8	7	5
(?) +	1.668	4	--	--
(004,021)	1.653	28	24	26
(022)	1.517	9	8	10

Sample 1.5-1 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(014)	1.488	tl	--	--
(113)	1.470	4	5	5
(?) +	1.360	4	--	--
(023)	1.350	5	--	--
$(114,121)_a$ + $(121)_b$	1.269 1.265	23	23	22
(015)	1.235	7	8	7
(122)	1.203	8	7	7
(030)	1.140	tl	--	--
(123)	1.115	5	--	--

Sample: 1.8-6 Analyzed composition: $ZrTe_{1.43}$

Sample color and texture: Brown powder sinter

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Lattice parameters: a_o = 3.953, c_o = 6.630 Å.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.266	tl	--	--
$(001)_a$ +	6.692	50	52	53
$(001)_b$	6.530			
(010) +	3.400	2	2	2
$(002)_a$ +	3.341	4	4	4
$(002)_b$	3.287	32	32	30
(011)	3.019	100	100	100
(?)	2.621	tl	--	--
(?)	2.454	tl	--	--
(012)	2.368	44	41	42
(003)	2.198	13	11	9
(110)	1.967	27	29	30
(111) +	1.885	tl	--	--
(?) +	1.869	tl	--	--
(013) +	1.849	20	22	26
(?)	1.829	tl	--	--
(112)	1.691	7	6	8
(004,021)	1.651	29	28	27
(022)	1.516	11	10	10
(014)	1.486	tl	--	--
(113)	1.468	6	4	6
(023)	1.349	8	6	5

Sample 1.8-6 (cont.)

Line	d (uncorr.) Å.	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
$(114,121)_a$ + $(121)_b$	1.267 1.262	25	21	22
(015)	1.234	7	8	7
(122)	1.202	7	8	8
(030)	1.139	6	--	--
(123)	1.114	6	--	--
(025)	1.048	9	--	--

Sample: 1.3-7 Analyzed composition: $ZrTe_{1.37}$

Sample color and texture: Brown powder sinter.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Lattice parameters: a_o = 3.952, c_o = 6.632 Å.

Comments: A small amount of free, or partially reacted, Zr was present in the chemical analysis sample.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
$(001)_a$ +	6.682	37	43	44
$(001)_b$	6.539			
(010) +	3.401	2	--	--
$(002)_a$ +	3.346	4	4	5
$(002)_b$	3.292	24	22	30
(011)	3.024	100	100	100
(?)	2.625	tl	--	--
(?)	2.452	tl	--	--
(012)	2.371	46	49	63
(003)	2.199	8	8	10
(110)	1.969	27	29	28
(111)	1.882	tl	--	--
(013)	1.850	20	22	13
(112)	1.692	8	6	8
(004,021)	1.652	26	26	26
(022)	1.516	11	8	14
(014)	1.486	tl	--	--
(113)	1.470	6	6	2
(?)	1.367	tl	--	--
(023)	1.350	8	7	6
(120)	1.290	tl	--	--

Sample 1.3-7 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(114,121)	1.267	23	25	26
(015)	1.234	8	8	9
(122)	1.203	8	7	9
(030)	1.139	tl	--	--
(123)	1.114	6	--	--
(025)	1.048	9	--	--

Sample: 1.4-7 Analyzed composition: $ZrTe_{1.35}$

Sample color and texture: Brown powder sinter.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Lattice parameters: a_o = 3.952, c_o = 6.632 Å.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.190	tl	--	--
$(001)_a$ + $(001)_b$	6.697 / 6.539	41	35	24
(010) +	3.397	2	2	2
$(002)_a$ + $(002)_b$	3.342 / 3.290	4 / 25	4 / 28	4 / 26
(011)	3.021	100	100	100
(?)	2.621	tl	--	--
(?)	2.451	tl	--	--
(012)	2.368	44	45	46
(003) +	2.198	10	11	9
(?)	2.178	2	1	1
(110)	1.967	26	27	26
(111)	1.888	tl	--	--
(013)	1.849	20	27	27
(112)	1.691	6	8	7
(004,021)	1.651	24	24	25
(022)	1.516	9	8	10
(014)	1.486	tl	--	--
(113)	1.468	6	5	6
(023)	1.349	7	6	8
$(114,121)_a$ + $(121)_b$	1.268 / 1.264	15 / 6	21 / 5	18 / 4

Sample 1.4-7 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
$(015)_a$ +	1.234	4	7	8
$(015)_b$	1.228	1	--	--
$(122)_a$ +	1.203	5	5	6
$(122)_b$	1.200	3	2	2
(030)	1.138	5	--	--
$(123)_a$ +	1.115	3	--	--
$(123)_b$	1.112	1	--	--
(025)	1.048	6	--	--

Sample: 1.0-8 Analyzed composition: $ZrTe_{1.33}$

Sample color and texture: Brown powder sinter plus some Blackish=brown material at the bottom.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Lattice parameters: $a_o = 3.957$, $c_o = 6.647$ Å.

Comments: Free, or partially reacted, Zr was present in the chemical analysis sample.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
$(001)_a$ +	6.755	4	3	4
$(001)_b$	6.622	19	17	24
$(002)_a$ +	3.362	5	3	4
$(002)_b$	3.313	10	7	9
(011)	3.040	100	100	100
(?)	2.569	12	--	--
(?)	2.458	13	14	13
$(012)_a$ +	2.404	6	6	6
$(012)_b$	2.380	36	32	36
(003)	2.209	4	--	--
(110)	1.975	35	36	37
(111) +	1.886	6	2	3
(013)	1.857	16	13	17
(112)	1.697	5	6	5
(004,021)	1.657	22	19	20
(?)	1.614	9	tl	4
(022)	1.521	10	9	9
(113) +	1.473	5	--	--
(?)	1.461	3	--	--
(?)	1.368	5	--	--

Sample 1.0-8 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(023)	1.352	7	--	--
(?) +	1.285	11	--	--
(121)	1.270	20	18	24
(015)	1.238	7	--	--
(122)	1.204	12	--	--
(030)	1.140	t1	--	--
(123)	1.115	8	--	--

Sample: 1.1-7 Analyzed composition: $ZrTe_{1.29}$

Sample color and texture: Dark brown powder sinter.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Lattice parameters: a_o = 3.952, c_o = 6.633 Å.

Comments: Free, or partially reacted, Zr was present in the chemical analysis sample.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
$(001)_a$ +	6.732	4	4	6
$(001)_b$	6.554	32	23	34
$(002)_a$ +	3.339	4	2	2
$(002)_b$	3.292	15	14	14
(011)	3.022	100	100	100
(?)	2.623	tl	--	--
(?)	2.446	4	tl	6
(012)	2.370	46	39	44
(003)	2.201	7	tl	8
(110)	1.968	31	31	32
(111)	1.885	tl	--	--
(013)	1.580	20	17	17
(112)	1.692	10	tl	6
(004,021)	1.652	25	19	21
(022)	1.516	11	10	12
(014)	1.486	tl	--	--
(113)	1.469	6	--	--
(?)	1.366	tl	--	--
(032)	1.350	6	--	--
$(114,121)_a$ +	1.268	18	17	17
$(121)_b$	1.264	5	4	4

Sample 1.1-7 (cont.)

Line	d (uncorr.) Å.	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(015)	1.234	8	--	--
(122)	1.202	8	8	10
(030)	1.139	tl	--	--
(123)	1.115	7	--	--
(025)	1.050	10	--	--

Sample: 1.7-6 Analyzed composition: $ZrTe_{1.19}$

Sample color and texture: Brown powder sinter with some darker brown material and some golden-brown material.

Sample tube color: Clear with remnants of a Te ring.

Firing schedule: 600°C for 130 hours.

Lattice parameters: $a_o = 3.967$, $c_o = 6.684$ Å.

Comments: A very small amount of free, or partially reacted, Zr was present in the chemical analysis sample. There was a very small amount of gold liquid-like material, but not enough for a chemical analysis sample.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.672	26	17	24
(010)? +	3.376	3	4	4
(002)	3.332	9	8	10
(011)	3.052	100	100	100
(?)	2.801	tl	--	--
(?)	2.713	tl	--	--
(?)	2.460	12	11	12
$(012)_a$ +	2.408	41	38	38
$(012)_b$	2.389			
(003)	2.220	tl.	--	--
(110)	1.983	35	34	37
(111) +	1.983	5	5	4
(013)	1.865	19	22	18
(112)	1.704	7	6	6
(004,021)	1.663	23	19	20
(022)	1.525	10	7	10
(114) +	1.279	20	20	23
(121)	1.272			

Sample 1.7-6 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(015)	1.242	tl	--	--
(122)	1.208	8	--	--
(030)	1.142	tl	--	--
(123)	1.120	6	--	--

Sample: 1.2-10 Analyzed composition: $ZrTe_{1.14}$

Sample color and texture: Dark brown powder sinter plus some free, or partially reacted, Zr.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Lattice parameters: a_o = 3.956, c_o = 6.666 Å.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
$(001)_a$ +	6.727	3	5	4
$(001)_b$	6.564	28	18	13
$(002)_a$ +	3.346	4	2	2
$(002)_b$	3.297	12	11	12
(011)	3.027	100	100	100
(?) +	2.477	2	--	--
(?)	2.449	4	--	--
$(012)_a$ +	2.397	4	3	3
$(012)_b$	2.372	40	37	32
(003) +	2.204	4	5	8
(?)	2.192	2	--	--
(110)	1.970	32	32	30
(111)	1.890	tl	--	--
(013)	1.852	16	14	21
(112)	1.694	6	tl	5
(004,021)	1.652	21	23	19
(022)	1.517	10	8	4
(014)	1.490	tl	--	--
(113)	1.471	tl	--	--
(?)	1.365	6	--	--
(023)	1.351	5	--	--
(114,121)	1.268	22	17	12

Sample 1.2-10 (cont.)

Line	d (uncorr.) Å.	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(015)	1.235	tl	--	--
(122)	1.204	8	--	--
(030)	1.140	tl	--	--
(123)	1.115	tl	--	--
(025)	1.050	tl	--	--

Sample: 0.9-7 Analyzed composition: $ZrTe_{0.94}$

Sample color and texture: Dark brown powder sinter with free, or partially reacted, Zr.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Lattice parameters: $a_o = 3.964$, $c_o = 6.646$ Å.

Comments: Peaks (014) and (113) were unresolvable, being one low, broad band.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
$(001)_a$ +	6.707	4	7	6
$(001)_b$	6.537	21	19	18
$(002)_a$ +	3.352	4	4	6
$(002)_b$	3.302	12	9	12
(?) +	3.059	8	11	11
(011)	3.024	100	100	100
(?) +	2.444	13	16	14
$(012)_a$ +	2.396	6	12	11
$(012)_b$	2.371	40	40	36
(?) +	1.983	8	8	7
(110)	1.968	32	40	34
(111) +	1.886	5	9	9
(013)	1.854	19	18	19
(112)	1.692	tl	--	--
(?) +	1.662	8	--	--
(004,021)	1.652	16	18	24
(022)	1.518	11	tl	11
(?)	1.365	9	tl	--
(023)	1.348	6	tl	--
(?) +	1.276	8	5	6

Sample 0.9-7 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities		
		Slow Scan	Count Scan	
		(Wt.%)	(Wt.%)	(Ct.%)
(114,121)	1.268	18	15	18
(122)	1.204	9	--	--

Sample: 0.8-7 Analyzed composition: $ZrTe_{0.67}$

Sample color and texture: Dark brown powder sinter with free, or partially reacted, Zr.

Sample tube color: Clear

Firing schedule: 600°C for 130 hours.

Lattice parameters: a_o = 3.970, c_o = 6.674 Å.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
$(001)_a$ +	6.712	2	2	3
$(001)_b$	6.564	14	17	20
$(002)_a$ +	3.347	3	5	5
$(002)_b$	3.306	7	6	6
(?)	3.205	6	13	13
(011)	3.031	100	100	100
(?)	2.642	tl	--	--
(?)	2.558	tl	--	--
(?) +	2.446	7	10	12
$(012)_a$ +	2.394	8	5	6
$(012)_b$ +	2.378	37	35	42
(?)	2.338	4	4	4
(003)	2.211	7	tl	9
(?) +	1.986	6	7	8
(110)	1.973	34	32	35
(111) +	1.886	4	5	5
(013)	1.857	17	12	14
(112)	1.697	6	tl	6
(?) +	1.666	6	--	--
(004,021)	1.655	13	20	20
(022)	1.522	10	tl	12
(?) +	1.364	4	--	--

Sample 0.8-7 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities		
		Slow Scan	Count Scan	
		(Wt.%)	(Wt.%)	(Ct.%)
(023)	1.354	6	--	--
(?) +	1.279	6	6	6
(114,121)	1.270	16	17	18

APPENDIX F

X-RAY RESULTS OF CHEMICALLY ANALYZED SAMPLES FIRED AT 817°C, THEN AT 600°C

Sample: 1.0-4 Analyzed composition: $ZrTe_{1.32}$

Sample color and texture: Black powder sinter.

Sample tube color: Black

Firing schedule: 817°C for 130 hours, then at 600°C for 130 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.622	3	tl	0
(001)	6.697	15	12	13
(?)	4.604	4	--	--
(?)	3.622	3	4	4
(010) +	3.404	4	5	4
(002) +	3.345	20	20	16
(?)	3.258	19	20	16
(011) +	3.063	100	100	100
(?)	3.002	15	16	16
(?) +	2.723	6	6	6
(?)	2.622	31	35	33
(?) +	2.535	10	11	10
(?) +	2.488	49	52	49
(?) +	2.454	4	6	5
(012) +	2.400	64	43	59
(?)	2.358	6	6	6
(003)	2.229	9	8	6
(?)	2.159	12	13	11

Sample 1.0-4 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	2.109	6	5	4
(110)	1.988	30	31	28
(?) +	1.921	18	22	18
(013) +	1.876	38	41	35
(?)	1.848	6	7	6
(?)	1.770	3	--	--
(020) +	1.710	9	9	8
(112)	1.698	3		
(004,021) +	1.670	25	26	24
(?)	1.658	6	--	--
(?)	1.628	tl	--	--
(022) +	1.530	24	24	20
(014) +	1.501	19	19	15
(113)	1.484	6	4	4
(?)	1.418	tl	--	--
(?) +	1.379	7	6	6
(?)	1.363	6	4	4
(023) +	1.346	10	9	7
(005)	1.331	4	3	2
(120) +	1.296	5	5	4
(114,121) +	1.279	28	26	21
(?) +	1.262	4	--	--
(015) +	1.247	11	10	8
(?)	1.235	3	--	--
(122) +	1.212	9	8	5
(?) +	1.205	4	8	6
(024)	1.195	10	13	9
(?)	1.173	11	11	8
(030)	1.148	9	11	6

Sample 1.0-4 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(031)	1.124	7	6	4
(032)	1.086	4	--	--
(016)	1.058	6	--	--
(025)	1.047	tl	--	--

Sample: 1.4-4 Analyzed composition: $ZrTe_{0.95}$

Sample color and texture: Light black powder sinter.

Sample tube color: Black

Firing schedule: 817°C for 130 hours, then at 600°C for 130 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.662	7	6	6
(?)	4.573	tl	--	--
(010) +	3.394	3	2	2
(002) +	3.334	19	19	19
(?)	3.254	14	16	16
(011)	3.0623	100	100	100
(?) +	2.722	47	40	45
(?) +	2.659	6	4	4
(?)	2.618	3	3	3
(?) +	2.531	2	--	--
(?) +	2.485	46	42	40
(?) +	2.438	4	7	7
(012)	2.400	28	25	24
(003)	2.219	6	4	4
(?)	2.108	13	10	11
(110) +	1.992	39	41	46
(?) +	1.962	4	4	4
(?) +	1.926	4	6	6
(013)	1.872	36	32	36
(112)	1.710	6	7	6
(004,021) +	1.669	17	18	18
(?)	1.659	9	6	6
(?) +	1.626	3	3	2
(?)	1.607	8	8	5

Sample 1.4-4 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(022)	1.530	7	6	4
(014) +	1.499	11	11	9
(113)	1.483	4	3	3
(?)	1.449	4	tl	2
(023) +	1.362	8	6	5
(?)	1.346	9	7	6
(114,121)	1.279	26	21	21
(015)	1.247	10	--	--
(024)	1.195	tl	--	--
(?)	1.172	10	9	8
(030) +	1.150	6	--	--
(?)	1.141	3	--	--
(031)	1.125	8	--	--
(032)	1.086	tl	--	--
(016)	1.056	tl	--	--

Sample: 0.8-4 Analyzed composition: $ZrTe_{0.84}$

Sample color and texture: Gray-brownish black powder sinter.

Sample tube color: Black

Firing schedule: 817°C for 130 hours, then at 600°C for 130 hours.

Comments: The most intense line is not (011).

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.570	24	19	14
(?)	5.262	13	tl	8
(?)	3.604	14	15	15
(010) +	3.391	9	13	12
(002) +	3.325	5	7	6
(?) +	3.246	130	120	113
(?)	3.157	32	31	30
(?)	2.996	100	100	100
(?) +	2.896	tl	tl	16
(?)	2.831	16	12	
(?)	2.740	15	15	14
(?) +	2.672	22	19	20
(?) +	2.617	212	200	212
(?) +	2.533	73	65	68
(?) +	2.483	268	241	256
(?) +	2.451			
(012) +	2.403	88	82	87
(?) +	2.351	45	44	46
(?)	2.298	8	8	9
(?) +	2.247	5	--	--
(003)	2.219	25	23	26

Sample 0.8-4 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?) +	2.156	84	83	74
(?) +	2.108	31	32	28
(?)	2.078	13	7	6
(?) +	1.918	110	96	84
(013) +	1.877	93	94	82
(?)	1.844	39	40	35
(?)	1.770	26	23	16
(020) +	1.713	16	24	17
(112)	1.698	20		
(004,021)	1.658	34	26	19
(?)	1.619	31	18	12
(022) +	1.527	69	72	56
(014) +	1.498	101	98	77
(113)	1.474	8	12	9
(?)	1.418	17	18	12
(?)	1.378	40	44	33
(023)	1.345	42	35	23
(?)	1.329	24	19	13
(?)	1.296	46	34	28
(122) +	1.204	25	37	26
(024)	1.194	52	48	34
(?)	1.172	61	52	37
(030)	1.147	19	--	--
(123)	1.116	tl	--	--
(?)	1.038	tl	--	--

Sample: 0.9-4 Analyzed composition: $ZrTe_{0.82}$

Sample color and texture: Brownish-black powder sinter.

Sample tube color: Grayish-black against sample, gray above sample.

Firing schedule: 817°C for 130 hours, then at 600°C for 130 hours.

Comments: There was a broad band of lines from 72.00° to 75.00° 2θ, the region of the (120), (114), and (121) lines.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	4.559	tl	--	--
(?)	3.601	tl	--	--
(010) +	3.383	4	6	6
(002) +	3.324	9	10	10
(?) +	3.248	76	94	94
(?) +	3.125	18	26	26
(011) +	3.048	100	100	100
(?)	2.986	12	12	12
(?) +	2.748	11	13	13
(?)	2.708	32	30	30
(?)	2.617	18	14	12
(?) +	2.483	211	248	233
(?) +	2.435	40	48	45
(012)	2.399	16	14	13
(?)	2.279	tl	--	--
(?)	2.156	tl	--	--
(?)	2.104	tl	--	--
(?) +	2.016	16	20	17
(110) +	1.993	10	12	10
(?) +	1.967	17	19	16

Sample 0.9-4 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?) +	1.950	19	20	18
(?) +	1.923	36	43	38
(013)	1.877	77	93	82
(112)	1.707	tl	--	--
(004,021) +	1.657	43	48	37
(?)	1.626	12	12	9
(?)	1.601	tl	--	--
(?) +	1.528	7	13	11
(022) +	1.519	6	--	--
(014) +	1.498	57	71	59
(113)	1.483	5	--	--
(?) +	1.380	9	10	7
(?) +	1.370	4	--	--
(023) +	1.359	7	8	5
(?)	1.346	48	59	41
(?)	1.259	10	--	--
(015)	1.243	11	--	--
(?)	1.231	9	--	--
(122) +	1.204	6	4	4
(024)	1.195	27	28	25
(?)	1.172	48	52	46
(032)	1.085	16	11	12
(?)	1.036	14	10	7

Sample: 1.2-4 Analyzed composition: $ZrTe_{0.68}$

Sample color and texture: Light black powder sinter.

Sample tube color: Black, mottled against sample.

Firing schedule: 817°C for 130 hours, then at 600°C for 130 hours.

Comments: There are two bands of low, unresolvable lines, one from 71.75° to 77.40° 2θ and the other from 79.05° to 80.85° 2θ. The former encompasses the (120), 114), (121), and (015) lines; the latter encompasses the (122) and (024) lines.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	4.547	tl	--	--
(010) +	3.384	4	6	6
(002) +	3.324	10	10	9
(?) +	3.234	34	43	43
(?) +	3.121	16	17	17
(011)	3.049	100	100	100
(?) +	2.761	6	7	8
(?) +	2.710	35	35	37
(?)	2.614	14	13	14
(?) +	2.485	40	41	46
(?) +	2.486	40	39	43
(012) +	2.393	7	5	6
(?)	2.345	6	6	6
(003)	2.222	5	6	5
(?) +	2.110	7	10	10
(?)	2.074	1	--	--
(?) +	2.019	20	20	19
(110) +	1.971	35	10	10
(?) +	1.954		28	27
(?)	1.927	11	12	12

Sample 1.2-4 (cont.)

Line	d (uncorr.) (A.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(013)	1.876	18	14	13
(?)	1.758	5	7	7
(020) +	1.728	3	--	--
(112) +	1.707	10	--	--
(004,021) +	1.662	22	--	--
(?) +	1.644	8	--	--
(?) +	1.626	6	--	--
(?)	1.606	7	--	--
(022)	1.526	8	7	6
(014) +	1.499	13	10	9
(113)	1.478	6	3	2
(?)	1.446	tl	--	--
(?)	1.417	tl	--	--
(?) +	1.377	12	--	--
(023) +	1.360	9	--	--
(?)	1.346	12	--	--
(?)	1.173	11	11	13

Sample: 1.1-4 Analyzed composition: $ZrTe_{0.62}$

Sample color and texture: Light black powder sinter with some glinty material.

Sample tube color: Black

Firing schedule: 817°C for 130 hours, then at 600°C for 130 hours.

Comments: The most intense line is not (011).

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.609	22	20	22
(?)	5.376	14	11	14
(?)	4.557	6	--	--
(?)	3.606	12	--	--
(010) +	3.394	10	9	8
(002) +	3.322	3	--	--
(?)	3.249	23	24	21
(?)	2.995	100	100	100
(?)	2.897	6	8	5
(?)	2.804	7	tl	6
(?)	2.738	11	13	8
(?) +	2.671	29	32	30
(?) +	2.617	226	223	211
(?) +	2.533	82	76	72
(?) +	2.481	92	77	73
(?) +	2.455	31	39	37
(012) +	2.403	88	90	85
(?)	2.353	34	31	30
(?)	2.298	10	9	7
(?)	2.265	tl	--	--
(003)	2.215	13	16	12
(?) +	2.156	85	84	77

Sample 1.1-4 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	2.107	31	28	25
(?) +	1.917	104	104	96
(013) +	1.876	51	54	50
(?)	1.844	34	35	33
(?)	1.770	25	24	17
(020) +	1.713	8	8	8
(112)	1.702	20	12	12
(?) +	1.670	4	--	--
(004,021)	1.658	8	--	--
(?)	1.619	8	--	--
(022) +	1.527	73	64	54
(014)	1.498	59	64	55
(?)	1.418	12	13	8
(?)	1.378	43	47	38
(023)	1.344	10	t1	6
(005)	1.329	18	18	15
(120)	1.296	38	33	32
(114,121)	1.271	10	--	--
(?) +	1.260	13	--	--
(?)	1.249	7	--	--
(015)	1.235	16	--	--
(122) +	1.204	36	40	30
(024)	1.193	46	40	31
(?)	1.173	21	19	15
(030)	1.147	22	--	--
(123)	1.116	6	--	--
(?) +	1.084	9	--	--
(032)	1.077	16	--	--
(025)	1.047	12	--	--

Sample: 1.3-4 Analyzed composition: --

Sample color and texture: Light black powder sinter.

Sample tube color: Gray against sample, black above sample.

Firing schedule: 817°C for 130 hours, then at 600°C for 130 hours.

Comments: The chemical analysis procedure was never completed; this was caused by the sudden appearance of large holes in the Pt crucible, just above the sample level.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
$(001)_a$ +	6.675	10		
$(001)_b$	6.650		9	8
(?)	4.571	2	--	--
(010) +	3.390	4	3	4
(002) +	3.330	22	18	18
(?)	3.251	4	4	4
(?) +	3.157	4	5	5
(011) +	3.057	100	100	100
(?)	2.974	2	2	2
(?) +	2.841	3	--	--
(?) +	2.718	43	35	38
(?)	2.628	4	3	3
(?) +	2.547	6	2	2
(?) +	2.484	14	14	12
(?) +	2.436	7	9	8
(012)	2.398	31	31	26
(?) +	2.3122	2	--	--
(003)	2.221	6	6	5
(?) +	2.106	11	10	10
(?)	2.069	1	--	--
(?) +	2.013	3	3	3

Sample 1.3-4 (cont.)

Line	d (uncorr.) Å.	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(110) +	1.989	37	39	37
(?)	1.960	10	10	9
(?)	1.925	tl	--	--
(111) +	1.899	2	2	2
(013) +	1.870	25	25	22
(?)	1.844	2	--	--
(112)	1.708	9	9	7
(004,021) +	1.667	26	23	20
(?)	1.648	3	4	3
(?)	1.606	6	6	4
(022)	1.530	7	6	5
(014) +	1.499	4	4	3
(113)	1.482	4	4	3
(?)	1.447	3	--	--
(?)	1.381	tl	--	--
(023) +	1.362	8	8	6
(?)	1.347	2	4	2
(120)? +	1.292	3	2	2
(114,121) +	1.278	26	26	22
(?) +	1.262	3	3	3
(015)	1.248	9	9	8
(122) +	1.214	4	--	--
(024)	1.199	4	--	--
(?)	1.172	4	5	4
(030)	1.148	5	--	--
(123)	1.124	8	--	--
(032)	1.087	tl	--	--
(016)	1.058	tl	--	--

APPENDIX G

X-RAY RESULTS OF CHEMICALLY ANALYZED SAMPLES FIRED AT 700°C

Sample: 1.4-5 Analyzed composition: $ZrTe_{1.51}$

Sample color and texture: Blackish-brown powder sinter.

Sample tube color: Clear above the sample, black against the sample.

Firing schedule: 700°C for 200 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.677	16	13	13
(?)	4.592	tl	--	--
(010)? +	3.385	4	3	3
(002)	3.336	17	18	17
(011)	3.054	100	100	100
(?) +	2.647	2	--	--
(?)	2.620	1	--	--
(?) +	2.475	3	4	4
(012)	2.395	52	56	53
(003) +	2.227	6	8	7
(?)	2.200	1	1	0
(110)	1.984	30	31	31
(111)	1.901	tl	--	--
(013)	1.869	20	20	20
(020,112)	1.706	6	6	5
(004,021)	1.666	25	26	24
(022)	1.529	11	13	11
(014)	1.502	2	3	2
(113)	1.482	4	4	3

Sample 1.4-5 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(023)	1.361	5	7	6
(114,121)	1.277	23	26	24
(015)	1.246	6	8	7
(122) +	1.211	9	11	9
(024)	1.198	1	--	--
(030)	1.147	4	5	4
(123)	1.122	5	6	6
(032)	1.084	tl	--	--
(016,025)	1.058	8	10	8

Sample: 1.1-5 Analyzed composition: $ZrTe_{1.22}$

Sample color and texture: Brownish-black powder sinter with a goldish tinge.

Sample tube color: Black

Firing schedule: 700°C for 200 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.692	10	10	9
(?)	4.573	tl	--	--
(010)? +	3.388	3	3	3
(002)	3.341	11	11	10
(011) +	3.059	100	100	100
(?) +	2.996	4	5	5
(?)	2.934	2	1	1
(?) +	2.656	2	--	--
(?)	2.620	3	--	--
(?)	2.534	2	--	--
(?) +	2.472	6	4	4
(012)	2.398	61	59	56
(003)	2.226	7	5	6
(110) +	1.986	36	32	31
(?)	1.962	5	4	4
(111) +	1.912	4	3	3
(013)	1.872	24	23	20
(020,112) +	1.708	10	8	7
(004,021) +	1.668	27	22	19
(?)	1.649	3	4	4
(022) +	1.530	16	17	13
(014) +	1.503	4	4	3
(113)	1.483	5	6	4

Sample 1.1-5 (cont.)

Line	d (uncorr.) Å.	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(023)?	1.363	7	7	5
(114,121) +	1.279	30	29	24
(?) +	1.263	2	--	--
(015)?	1.247	8	7	6
(122) +	1.213	11	11	9
(024)?	1.204	4	3	2
(030)	1.148	5	7	4
(123)	1.124	7	6	6
(032)	1.086	tl	--	--
(016) +	1.058	13	9	7
(025)	1.047	4	--	--
(?) +	1.032	3	--	--
(?)	1.025	4	--	--

Sample: 1.3-5 Analyzed Composition: $ZrTe_{1.15}$

Sample color and texture: Brownish-black powder sinter.

Sample tube color: Black

Firing schedule: 700°C for 200 hours.

Comments: Free, or partially reacted, Zr in chemical analysis sample.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Ct.%)	Count Scan (Ct.%)
(001)	6.730	8	7	6
(?)	4.578	tl	--	--
(010) +	3.407	4	2	3
(002)	3.355	11	10	10
(011) +	3.073	100	100	100
(?) +	3.016	11	15	15
(?)	2.944	4	2	2
(?) +	2.724	2	--	--
(?) +	2.669	3	--	--
(?)	2.627	4	--	--
(?) +	2.569	5	2	2
(?) +	2.538	3	3	3
(?) +	2.483	7	7	7
(012)	2.408	67	63	63
(?) +	2.261	2	--	--
(003) +	2.233	5	6	6
(?)	2.208	2	--	--
(?)	2.163	tl	--	--
(110) +	1.994	35	30	31
(?)	1.967	11	9	9
(?) +	1.920	5	1	2
(111) +	1.899	4	3	3

Sample 1.3-5 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(013) +	1.877	24	20	20
(?)	1.852	2	1	1
(020,112) +	1.713	9	6	6
(004,021) +	1.674	26	22	22
(?)	1.650	6	2	2
(022) +	1.534	17	15	14
(014) +	1.507	5	3	2
(113)	1.488	6	4	4
(?)	1.465	tl	--	--
(?)	1.420	tl	--	--
(?)	1.404	tl	--	--
(023)?	1.366	12	7	6
(?) +	1.295	1	--	--
(114,121) +	1.282	29	22	24
(?) +	1.264	3	--	--
(015)	1.250	8	5	5
(122) +	1.216	12	8	9
(024)	1.204	4	2	3
(030)	1.150	6	5	4
(123)	1.126	8	5	6
(032)	1.087	tl	--	--
(016,025)	1.061	8	5	5

Sample: 1.2-5 Analyzed composition: $ZrTe_{0.98}$

Sample color and texture: Brownish-black powder sinter with some glints of gold color.

Sample tube color: The top one-quarter was clear; the rest was black.

Firing schedule: 700°C for 200 hours.

Comments: Free, or partially reacted, Zr in chemical analysis sample.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	9.349	tl	--	--
(001)	6.722	13	11	11
(?)	4.585	tl	--	--
(010) +	3.407	3	3	2
(002)	3.350	17	16	15
(011) +	3.066	100	100	100
(?) +	3.004	4	5	5
(?)	2.942	3	1	1
(?) +	2.715	2	--	--
(?) +	2.662	2	--	--
(?)	2.625	3	--	--
(?) +	2.538	3	2	2
(?) +	2.483	5	5	4
(012)	2.403	65	66	62
(003) +	2.232	7	6	6
(?) +	2.202	2	1	1
(?)	2.165	1	--	--
(110) +	1.991	32	32	28
(?)	1.968	9	7	6
(?) +	1.926	2	2	2

Sample 1.2-5 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(111) +	1.908	3	2	1
(013)	1.874	28	26	22
(020,112) +	1.711	8	7	6
(004,021)	1.670	28	30	25
(022) +	1.531	17	16	13
(014) +	1.505	4	4	3
(113)	1.486	6	5	4
(023)	1.364	8	6	5
(114,121)	1.280	29	26	20
(015)	1.248	8	6	5
(122) +	1.214	8	11	8
(024)	1.207	4	3	2
(030)	1.148	5	6	3
(123)	1.124	7	8	5
(032)	1.086	tl	--	--
(016)	1.060	9	9	6

APPENDIX H

X-RAY RESULTS OF CHEMICALLY ANALYZED SAMPLES FIRED AT 650°C

Sample: 1.4-6 Analyzed composition: $ZrTe_{1.96}$

Sample color and texture: Golden-brown powder sinter with some free Te crystals.

Sample tube color: Clear

Firing schedule: 650°C for 500 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	7.347	tl	--	--
(001)	6.634	58	51	49
(?)	5.005	tl	--	--
(010)? +	3.369	4	5	4
(002)	3.311	51	52	49
(011)	3.040	100	100	100
(?)	2.885	tl	--	--
(?)	2.694	2	--	--
(?)	2.634	2	--	--
(?) +	2.471	4	3	3
(012) +	2.379	51	52	50
(?)	2.342	2	2	2
(003)	2.207	20	19	18
(?)	2.055	tl	--	--
(110)	1.975	30	28	26
(111) +	1.892	2	2	2
(013) +	1.855	27	26	24
(?)	1.823	2	2	2
(?)	1.779	tl	--	--

Sample 1.4-6 (cont.)

Line	d (uncorr,) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(112)	1.696	8	7	6
(004,021)	1.655	37	38	34
(022)	1.519	11	11	9
(014) +	1.490	4	4	3
(113)	1.472	8	7	6
(023)	1.352	7	6	6
(114,121)	1.268	26	26	23
(015)	1.235	11	11	9
(122)	1.204	9	9	7
(024)	1.189	1	--	--
(030)	1.140	4	4	4
(123)	1.115	7	7	5
(032)	1.078	tl	--	--
(025)	1.049	12	11	9

Sample: 1.3-6 Analyzed composition: $ZrTe_{1.42}$

Sample color and texture: Brown powder sinter with a small amount of blackish-brown material at the top and bottom of the sample.

Sample tube color: Clear, but with a light coating of black dust.

Firing schedule: 650°C for 500 hours.

Comments: There was some free, or partially reacted, Zr in the chemical analysis sample.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.664	14	15	15
(010) +	3.381	3	3	3
(002)	3.332	16	15	16
(011)	3.053	100	100	100
(?)	2.929	tl	--	--
(?)	2.796	tl	--	--
(?) +	2.646	2	--	--
(?)	2.618	1	--	--
(?)	2.461	6	4	4
(012)	2.392	57	56	53
(003) +	2.223	7	6	5
(?)	2.199	2	1	1
(?)	2.158	tl	--	--
(110)	1.982	33	32	30
(111) +	1.898	3	1	1
(013)	1.866	26	21	22
(112) +	1.703	8	7	7
(004,021)	1.664	27	25	22
(022) +	1.526	14	12	11
(014) +	1.499	3	3	3
(113) +	1.479	6	5	5

Sample 1.3-6 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	1.461	2	1	1
(023)	1.359	8	7	6
(114,121)	1.275	25	27	23
(015)	1.243	8	6	6
(122) +	1.209	11	10	9
(024)	1.196	1	1	1
(030)	1.146	5	4	4
(123)	1.120	6	6	5
(016)	1.056	11	9	7

Sample: 1.2-6 Analyzed composition: $ZrTe_{1.29}$

Sample color and texture: Dark brown powder sinter plus some free, or partially reacted, Zr.

Sample tube color: Black, except for a narrow band of clear at the top.

Firing schedule: 650°C for 500 hours.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.722	15	9	10
(010) +	3.400	4	6	4
(002)	3.346	12	13	12
(011)	3.064	100	100	100
(?) +	2.463	4	8	8
(012)	2.401	59	59	61
(003)	2.232	6	6	9
(110)	1.990	33	38	34
(013)	1.873	20	21	18
(112)	1.710	7	7	6
(114,021)	1.670	24	26	23
(022)	1.531	13	16	12
(014) +	1.502	2	tl	--
(113)	1.485	4	tl	--
(?)	1.419	tl	--	--
(023)	1.364	4	8	8
(114,121)	1.279	24	26	26
(015)	1.247	10	10	10
(122)	1.214	12	10	21

Sample: 0.9-6 Analyzed composition: $ZrTe_{1.03}$

Sample color and texture: Brown powder sinter.

Sample tube color: Black

Firing schedule: 650°C for 500 hours.

Comments: This sample was opened in a N_2 atmosphere.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.627	8	--	--
(?)	6.163	tl	--	--
(?)	5.867	tl	--	--
(010) +	3.385	6	--	--
(002)	3.348		7	8
(?)	3.247	6	8	9
(011)	3.046	100	100	100
(?)	2.931	tl	--	--
(?)	2.714	4	--	--
(?) +	2.528	6	5	5
(?) +	2.483	20	24	26
(?) +	2.451	13	18	19
(012)	2.400	50	54	59
(110) +	1.986	13	12	12
(?)	1.956	26	36	36
(111) +	1.894	5	5	5
(013)	1.873	10	13	15
(004,021) +	1.665	11	--	--
(?)	1.644	6	--	--
(022)	1.525	6	--	--
(023) +	1.363	6	--	--
(?)	1.348	3	--	--

Sample 0.9-6 (cont.)

Line	d (uncorr.) Å.	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(121)	1.280	7	--	--
(?)	1.260	5	--	--

Sample: 1.0-6 Analyzed composition: $ZrTe_{0.91}$

Sample color and texture: Black powder sinter.

Sample tube color: Black

Firing schedule: 650°C for 500 hours.

Comments: This sample was opened in a N_2 atmosphere.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.697	16	9	13
(010) +	3.395	2	3	4
(002) +	3.344	13	11	15
(?)	3.265	4	3	4
(011)	3.062	100	100	100
(?)	2.627	tl	--	--
(?) +	2.485	26	18	22
(?) +	2.457			
(012)	2.401	57	50	58
(003)	2.228	5	--	--
(110) +	1.990	28	24	29
(?)	1.964	7	8	9
(013)	1.875	20	19	24
(112)	1.711	6	--	7
(004,021)	1.669	22	18	24
(022)	1.530	12	7	13
(014) +	1.502	4	--	--
(113)	1.485	4	--	--
(023) +	1.365	7	9	12
(?)	1.348	3	2	3
(114,121)	1.280	19	15	23
(015)	1.248	4	tl	6
(122)	1.214	7	tl	8

Sample 1.0-6 (cont.)

Line	d (uncorr.) Å.	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(024)	1.199	tl	--	--
(030)	1.148	3	--	--
(123)	1.125	7	--	--
(016)	1.059	4	--	--

Sample: 0.8-6 Analyzed composition: $ZrTe_{0.87}$

Sample color and texture: Brown powder sinter.

Sample tube color: Black

Firing schedule: 650°C for 500 hours.

Comments: This sample was opened in a N_2 atmosphere.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001) +	6.672	8	--	--
(?) +	6.445	6	--	--
(?) +	6.134	5	--	--
(?)	5.861	6	--	--
(010) +	3.386	3	--	--
(002) +	3.346	10	9	13
(?)	3.256	6	6	8
(?) +	3.133	2	--	--
(011) +	3.048	100	100	100
(?) +	3.006	12	13	13
(?)	2.928	12	9	9
(?) +	2.717	4	--	--
(?)	2.668	4	--	--
(?) +	2.531	8	8	9
(?) +	2.484	11	12	9
(?) +	2.457	8	10	8
(012)	2.396	64	65	68
(003)	2.196	4	--	--
(110) +	1.989	18	16	16
(?)	1.961	33	34	37
(111) +	1.896	4	4	5
(013)	1.872	18	14	20
(004,021) +	1.665	11	--	--

Sample 0.8-6 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	(Ct.%)
(?)	1.644	6	--	--
(022)	1.527	13	--	--
(023) +	1.363	8	--	--
(?)	1.347	6	--	--
(114,121)	1.278	15	--	--
(?)	1.263	5	--	--

APPENDIX I

X-RAY RESULTS OF CHEMICALLY ANALYZED SAMPLES FIRED AT 600°C FOR 645 HOURS, WITH GRINDING AFTER 145 HOURS

Sample: 0.75-2 Analyzed composition: $ZrTe_{0.83}$

Sample color and texture: Black powder.

Sample tube color: Clear

Firing schedule: 600°C for 645 hours, with grinding after 145 hours.

Comments: The material was a brown powder sinter after the 145 hour firing at 600°C. The material was ground in a N_2 atmosphere and turned black before it was resealed in a fused silica tube. The powder did not sinter during the 500 hour firing.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.585	19	16	19
(010) +	3.375	4	3	4
(002)	3.319	14	11	12
(011) +	3.042	100	100	100
(?)	2.998	8	7	7
(012) +	2.388	54	53	51
(?)	2.367	6	4	4
(003) +	2.223	3	--	--
(?)	2.195	2	--	--
(110) +	1.982	35	35	46
(?)	1.960	7	6	8
(111) +	1.884	1	4	5
(013) +	1.866	15	18	22
(?)	1.820	4	7	8

Sample 0.75-2 (cont.)

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(112)	1.705	4	tl	6
(004,021)	1.651	24	21	16
(022)	1.528	12	11	17
(113)	1.482	4	--	--
(023)	1.361	10	--	15
(114,121)	1.278	24	23	32
(015)	1.246	6	--	--
(122)	1.212	9	7	10
(030)	1.148	5	--	--
(123)	1.124	6	--	--
(016)	1.060	tl	--	--

Sample: 0.67-2 Analyzed composition: $ZrTe_{0.69}$

Sample color and texture: Black powder plus free, or partially reacted, Zr.

Sample tube color: Clear

Firing schedule: 600°C for 645 hours, with grinding after 145 hours.

Comments: The material was a brown powder sinter with some free, or partially reacted, Zr after the 145 hour firing at 600°C. The material was ground in a N_2 atmosphere and turned black before it was resealed in a fused silica tube. The powder did not sinter during the 500 hour firing.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(001)	6.578	11	--	--
(?)	5.783	6	--	--
$(002)_a$ +	3.352	4	tl	--
$(002)_b$	3.314	8	tl	--
(011) +	3.033	100	100	100
(?) +	2.998	19	28	28
(?)	2.909	6	6	6
(?)	2.512	tl	--	--
(?) +	2.439	13	15	14
(012) +	2.370	52	49	47
(?)	2.366	12	16	15
(110) +	1.979	29	29	34
(?)	1.956	20	21	25
(?) +	1.882	4	--	--
(013)	1.863	14	16	20
(004,021) +	1.662	18	--	--
(?)	1.638	4	--	--
(022)	1.526	9	--	--
(114,121)	1.276	16	--	--

Sample: 0.50-2 Analyzed composition: $ZrTe_{0.21}$

Sample color and texture: Black powder with free, or partially reacted, Zr.

Sample tube color: Clear

Firing schedule: 600°C for 645 hours, with grinding after 145 hours.

Comments: The material was a brown powder sinter with some free, or partially reacted, Zr after the 145 hour firing at 600°C. The material was ground in a N_2 atmosphere and turned black before it was resealed in a fused silica tube. The powder did not sinter during the 500 hour firing. The intensities of the majority of the lines were too low to cut and weigh, so d-spacings and qualitative relative intensities are listed. No count scan was run.

Line	d (uncorr.) (Å.)	Relative Intensities Slow Scan (Wt.%)	Count Scan (Wt.%)	Count Scan (Ct.%)
(?)	3.246	small		
(011) +	3.045	med.		
(?)	2.996	small		
(?)	2.792	small		
(?)	2.616	small		
(?)	2.570	small		
(?)	2.483	med.		
(?)	2.454	strong		
(012)	2.403	small		
(?)	2.156	small		
(?)	1.958	small		
(?)	1.916	small		
(?) +	1.892	small		
(013)	1.877	small		
(?)	1.616	small		
(?)	1.467	small		

APPENDIX J

CALCULATIONS

A. Chemical Analysis Calculations

The amount of sample undergoing chemical analysis was determined by weighing the Pt crucible plus sample immediately upon transfer of the telluride to the crucible. The Pt crucible had been weighed previously. Just prior to transferring the telluride to the Pt crucible, the telluride was weighed in its Al crucible. The net sample weight was obtained by the following equation:

$$\text{net sample weight (g.)} = \text{wt. of crucible + sample (g.)} - \text{wt. of cruc. (g.)}.$$

If any material was lost during the transfer, a correction was applied to the initial "dry" weight of the sample. This procedure was necessary, because prior to analysis the sample was exposed to the atmosphere and gained weight (see Section IV-E). The equation used in this calculation was:

$$\text{corr. "dry weight" (g.)} = \text{initial dry weight (g.)} \times \frac{\text{net sample wt. - wt. Pt cruc. (g.)}}{\text{net sample wt. - wt. Al cruc. (g.)}}$$

The residue from the chemical analysis procedure was ZrO_2. The amount of Zr in a given amount of ZrO_2 was obtained by the following equation:

$$\text{equiv. wt. Zr (g.)} = \text{wt. } ZrO_2 \text{ (g.)} \times \frac{\text{atomic wt. of Zr (g.)}}{\text{molecular wt. of } ZrO_2 \text{ (g.)}}.$$

The Te content of the sample was calculated from the

equation:

wt. Te (g.) = corr. "dry weight" (g.) - equiv. wt. Zr (g.).

The Te/Zr mole ratio was calculated from the following equation:

$$\frac{\text{moles Te}}{\text{moles Zr}} = \frac{\text{wt. Te (g.)/atomic wt. Te (g.)}}{\text{wt. Zr (g.)/atomic wt. Zr (g.)}} .$$

B. Weight Gain Calculations

The percent weight gain was calculated from:

$$\text{percent weight gain} =$$

$$\left[\frac{\text{wt. of sample + crucible - wt. of crucible}}{\text{initial net sample weight}} - 1.0000\right] \times 100.$$

This percent weight gain value was converted to "moles of water gained" by the following equation:

$$\text{moles of water gained} =$$

$$\frac{\text{percent weight gained}}{100} = \frac{\text{mol. wt. of } ZrTe_x}{\text{mol. wt. of } H_2O^x},$$

where x = Te/Zr mole ratio.

APPENDIX K

X-RAY RESULTS OF YEAR-OLD 600°C SAMPLES

Line	Te	Sample 2.0-3	1.8-3	1.4-3
	3.230	3.238	3.236	3.238
(011)			3.040	3.044
			2.433	
(012)			2.378	2.378
	2.351	2.351	2.351	2.351
	2.228	2.229	2.229	2.229
	2.087	2.086	2.086	2.085
	1.980	1.976	1.975	1.974
	1.835	1.834	1.835	1.835
	1.781		1.781	1.782
	1.758		1.759	
(021)			1.656	
	1.616	1.617	1.617	1.617
	1.479	1.478	1.477	1.478