FINAL REPORT

Contract NAS8-33548

August 1979 - October 1981

X-RAY DIFFRACTION ANALYSIS OF Nb_3Ge AND NbGe ALLOYS

by

Jack H. Davis

and

K. W. House

Prepared by

National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama 35812

Submitted by

The University of Alabama in Huntsville
School of Mathematical and Natural Sciences
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I. ACKNOWLEDGEMENTS

The authors wish to acknowledge Dr. L. L. Lacy's conception of the drop tube superconductor processing experiment. His interaction and collaboration with top laboratories across the country has indeed enhanced the local solid state scientific community.

The following work was performed over a two-year period by Graduate Research Assistant Mr. K. W. House under the guidance of Associate Professor J. H. Davis. Although this document is the final contractual report on this project, Mr. House's upcoming thesis should indeed be referenced for further scientific information.

The efficient clerical assistance of Mrs. Traudel von Spakovsky and Mrs. Joan Broughton are gratefully acknowledged. Both Mr. Thomas Rathz and Mr. Michael Robinson provided close technical and scientific interaction.

II. INTRODUCTION

Input all along on this project has been on an almost continuous basis. Mr. House provided direct input as an integral part of the NASA Team. Also a NASA authored journal publication[1] references the input of Mr. House's. In fact the above article on metastability hinges upon his annealing induced shifts in the alpha (A-2) phase lattice constant.

The reader may find this input somewhat crypic and sometimes without conclusions. This effort was a small part of a larger NASA program and must be viewed in that context.

This high volume report is rather rough, as it is a composite of the three earlier reports attached together in chronological order to form Appendices A, B, and C.

III. OBJECTIVE AND RESULTS

Although our objectives (qualitative phase identification and accurate lattice constant determination) were met in this effort, we were admittedly hoping to generate a scientific, technical breakthrough: (the drop tube) synthesis and x-ray identification of (high T_c) stochiometric bulk Nb_3Ge. Of all the A-15 samples of NbGe alloy examined, DT 094 is unique in that it was at least 99% pure A-15 phase. Also its diffraction peaks were noisy as if there were about a one percent compositional variation on this phase. DT 094, however, was only a large fragment of the drop tube drop, and thus its small sample size may have reduced the intensity, thus enhancing fluctuations enough to explain some of the loss of peak resolution.

IV. DISCUSSION

One possible explanation for the apparent undetectability of up to 20% of high metastable (a = 0.5166 nm) region, could be due to the possibility of a continuous spreaded of A-15 lattice constants in the 0.514 to 0.516 nm range, thus generating a smear of diffraction noise just to the left of each of A-15 peaks. Unfortunately, the K alpha 1 peak associated with the small fraction of metastable A-15 phase (a = 0.5155 nm) would fall right on top of the K alpha 2 peak of the stable phase (a = 0.5168 nm), thus further contributing to difficulty of detection. A precision monochrometer to diffract off the unwanted K alpha 2 line would help. Perhaps this K alpha 2 line may be analytically by the so-called Ratchinger[3] technique subtracted. Along a similar line of reasoning, Mathias[4] may have been forced to use the Debye Scherrer technique by virtue of having a much smaller sample volume than in the present investigation.

V. SUGGESTIONS

As the NbGe phase diagram does not lend itself to the formation of single phase A-15, however, the drop tube seems to have overcome this problem at least as far as DT 094 is concerned. This idea in fact may be patentable technique for producing pure (but still unstochiometric) single phase A-15. Splat-cooling rates offer great advantages and the drop tower offers good undercooling advantages in super-cooling by about 400 K. So intergrating a cryogenic splat-cooler to the bottom of the drop tube may produce more metastability in phases such as A-15. It appears that success in generating the bulk metastable phase may lie with smaller samples (0.1 mm) whose dimensions lie closer to those of the micron thick, superconducting stochiometric, high T_c Nb_3Ge thin films. These smaller superconducting volumes will require a greater inductances bridge sensitivity. The present 20% coil unbalance should be compensated for by a variable mutual inductor coupling the primary to the secondary detector circuit.

REFERENCES

1. Lacy, L. L., et al., Jour, Appl. Phys. V 53, P. 682, January 1982.

2. Rathz, T. J. NASA TM 82399, January 1981.

3. Ratchinger, W. A., Jour. Sci. Inst. 25, P. 254, 1948.

4. Mathias, B. J., et al., Phys. Rev. V. 139, No. 5A, P. A1501, 1965.

APPENDIX A

PROGRESS REPORT I

Contract NAS8-33548

August 1979 - January 1980

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February 1980

Official work on this task began September 1st. when Mr. Kenneth House, a new 1/2 time graduate research assistant with undergraduate experience in x-ray diffraction, began a 1 month literature of review of "Nb_3Ge" with emphasis on x-ray diffraction. A chronological chart of the "Nb_3Ge" findings (through 1978) is shown in Tables 1,2,3,4,5,6, and 7. During October the Phillips x-ray diffractometer with new Ortec counting electronics was calibrated using Silicon, Niobium, and Quartz powder samples. The signal to noise ratio was optimized by adjusting the detector voltage, the amplifier gain, and the upper and lower level discriminators. The resulting I vs. 2θ scan for quartz yields a trace (Fig. 1) similar to the ideal "Text Book" example but with a 5 times better signal to noise ratio.

Good resolutions of the $K\alpha_1$ and $K\alpha_2$ peaks at an angle as low as 50° (2θ) indicates a good resolving power for the total system even in this fast scan mode (1°/min). The peak is seen to fall within 0.02° of the handbook valued of 26.44° and 20.83°. At slower scan speeds the .02" should reduce to ~.01°.

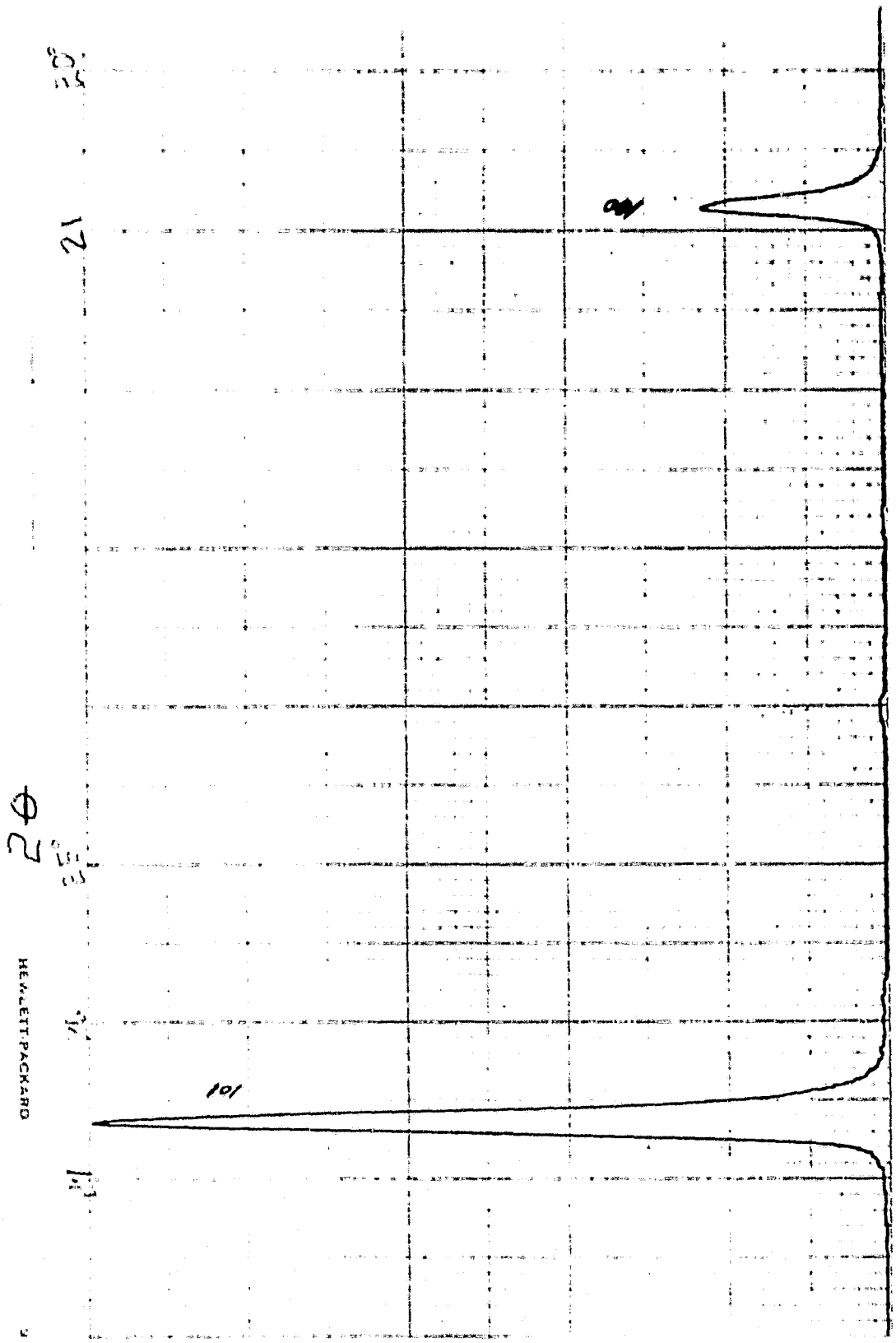

Figure 1: Portion of the Quartz calibration (1°/min.) scan showing the (100) 2θ =20.83° peak and the (101) 2θ = 26.64° peak. Full-scale is 3000 cps with a 1SEC time-constant.

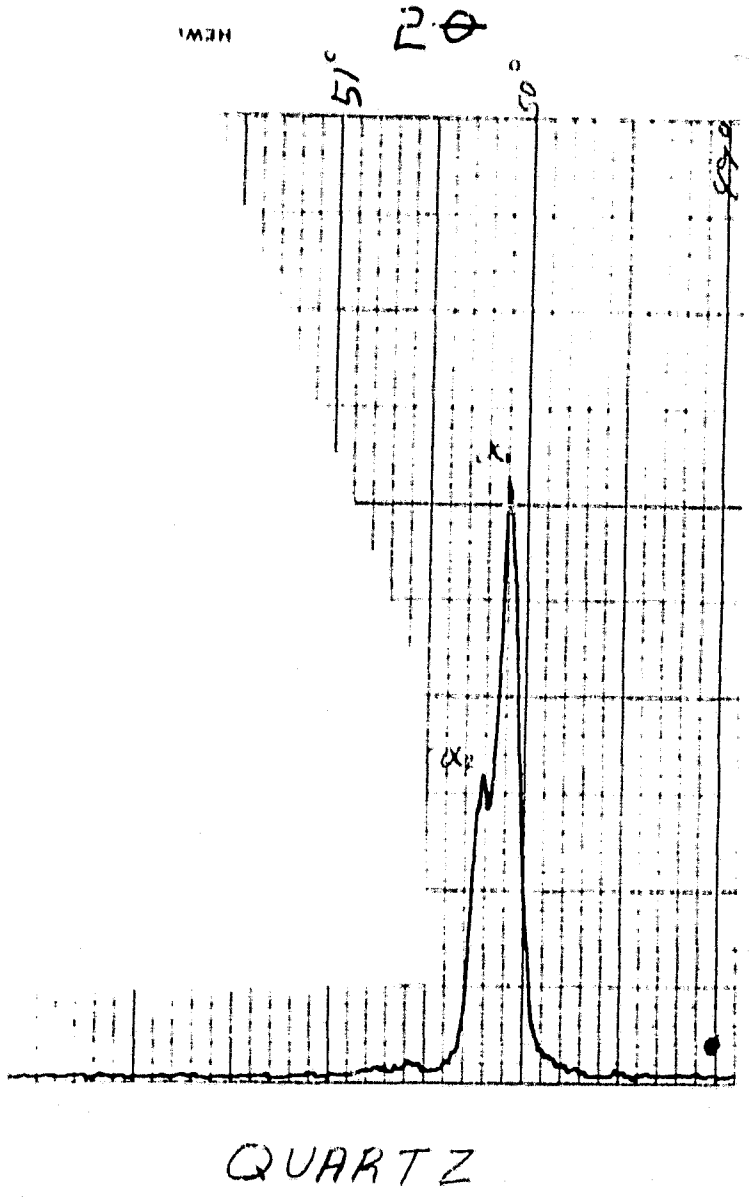

Figure II The 2θ range around 50° is shown at 1000 CPS full scale at 1°/minute with a 1 second time-constant. The good resolution of the α_1 and α_2 peak at a 2θ angle as low as 50° indicates good resultion by the system.

PROPERTIES OF Nb_3Ge

No.	Year	Author	STOICH./ST.ICH.	Bulk/ Film/Thick	Tc °K	Density gm/cm³	Lattice Parameter A°	S IRO Parameter	Resistivity Ratio & Hardness	J_c A/cm²	Additional Misc., etc.
1	19[illegible]	Reed, Gates	Nb_3Ge/No	Bulk	S						
2	63	Carpenter	Nb_3Ge/Yes	Bulk		8.7 ± 0.5	5.[illegible] ± .002				data, de… enthalpy, entropy, Press
3	1963	Matthias	3:1/?	Bulk	6.90		5.165				
4	65	Matthias	3:1/Yes	Bulk?	17		Extrapolated 5.15±0.01	0.4 ± 0.15			
6	68	Otto (Germany)	3:1/?	?	<5		..5.17		1700Kp/mm²		
8	70	Hanak	3:1/?	Film 3,000 to 10,000 A°							
9	70	Mueller (German)									
11	71	Brelko (USSR)									elec.re-sistivity magnetic suscepti-bility
12	71	Hallais (French)	"the move ordered → higher Tc"							at 4.2K* 80 kOe	
13	72	Kiwake (Japan)	$(Nb_{.95}Ta_{.05})_{.85}Al_{.15}Ge_{.15}$	Bulk-Rod	21					1.2 x 10⁴ A/cm²	
17	73	Gavaler	3:1/Yes ?	Film (?)	~22			Sputtering			"perfect Stoich => higher Tc"

Table 1 Properties of "Nb_3Ge"

20	1974	Mihailov (USSR)	3:1/	Bulk (?)	~22.3 >22			resistance & mag. measure ments to determine T_c
21	74	Cavaler	3:1/Yes?	Film 3 - 20x10^3 A°		P.D. 5.143 ± 0.003 5.152 ± 0.001 D.S. Camera	18.0K zero field ~10^5 A/cm	4-point resistive technique to measure T_c ± 0.1K
22	1974	Testardi	3:1/?	Film	23.2 ± 0.2			
23	74	Chencinski; Cadieu	3:1/	Film	>21			RF Sputtering
24	74	Vieland	3:1/ at 9. Nb ?	Film (Thick) ?	20.6	5.25	4.2K 200 KG ~10^5	CVD
25	74-5	Ga aler	3:1/	Film (5-50μm)	>21.5			
26	75	Testardi	3:1/Yes	Film	>23	5.13-.14 film 5.17 Bulk	P300K/025K resistivity ratios made	4.2K 200 KE ~10^5
27	75	Newkirk; Valencia	3:1/	CVD Bulk (.02-.06 mm	~21	5.146 ±0.002	@13.8k ± $1.78x10^6$	Resistence measure of T_c susceptness of T_c
28	75	Ghosh	3:1/Yes	Sputtered film 0.5 to 2.0 μm	~21	5.142 ±.003	Zero appld. mag. field.	T_c ~10°K
29	75	Bartlett	3:1/?	Film 10-60μm CVD			1.6×10^6	
31	7-	Cadieli, Chencinski	3:1/?	Sputtering Film ?	~22			
33	75	Newkirk	3:1/	CVD Film ?	21.2			Heat capacity from 3-32°K No anomalies up to 65°K.
34	76	Webb;Englehart	3:1/?	CVD ?	≤ 23.0 ± 0.2			Gas composition affects T_c, a_o

Table 2 Properties of "Nb_3Ge"

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No.	Year	Author	Nb:Ge [illegible]	Bulk/Film/Thick	T_c °K	Lattice Parameter Å	J_c A/cm²	Additional Misc., etc.
36	'76	Ageev (Russian)	3:1	Anneal [illegible]	~7.2			[illegible] annealing increases order for Nb_3Al [illegible]
37	'76	Roy; Rogowski	3:1 ?	Sputt/film/2--				Disorder & strain from mech. polishing slightly lowers T_c (Annealing restores)
41	'76	Webb, Aravenius	3:1 ?	CVD film thick	23.0			
43	'76	Biesbee	3:1	e-evaporation film CVD	>21.5			[illegible] [illegible]
45	'76	Newkirk, Valencia	3:1	film	–	–		T_c & a_c as a function of annealing & irradiation by neutrons (annealing restored T_c & a_c after)
46	'75	Vereshchagin (USSR)	3:1	High pressure High temp. bulk	19.4 70KB (1450°C)	5.135 – .140		
47*	'76	Roy; Rogowski	3:1	Sputter of films [illegible]	21.5	5.131		EMP predicts a_c = 5.127 T_c = 23.4 for Stoich.
51	'74	Zagryazhskii	3:1	?	high?	5.145		
52*	'76	Rogowski	3:1 No Emp	Films (~.56 μm)	21.2 19.7	5.178 5.142	Coarse grains	EMP to determine Nb/Ge ratio T_c measures as approach stoich. 5.54 ± 0.08 3.61 ± 0.54
55	'76	Braginski	5:5 – 3:1	NaCVD film thick			≃ 10^6	
59	'77	Testardi	3:1	Thin film				depend of T_c resist. ratio, & phase on stoich. (Stoich. is not the most important factor in affecting T_c.)

Table 3   Properties of "Nb_3Ge"

61	'77	Newkirk, Valencia	5:3 - 3:1/	CVD	~21		2.5 x 10^6 A/cm^2	Temp. dependence of $J_c(T) = J_c(0)\ 1 - \frac{T}{TC}^2$
63	'77	Newkirk, Valencia	3:1					Structural quality improved addition of ~5 at % O
66	'77	Sigsbee	3:1/	Coevaporation	>21			Enhanced Superconductivity of addition of O during growth
67	'77	Saur; Haenssler (German)	3:1/	d.c. sputtering 0.06-1.5μm	≤22.7	5.14		
68	'77	Kudo - Japan	3:1	coevaporation 2000A° 50A°	22 4.2			Nb_3Ge grains ~500A° in diameter
69	'76	Carlson, Los Alamos	3-1 & 5-3	CVD Bulk ?	21		2.5 x 10^6	2-3% of 5-3 phase present J_c measured @ 21 +4°K
70	'77	Testardi	3-1	Varied ?				Measured temp. dep. of elec. resistivity of 3-1 various samples
71	'77	Webb	?	?				Resistances are linear functions of T^2 from Tc to 40°K Also lattice heat capacities
73	'77	I Lona	3-1/Yes	E.B.Dep.films/?	21.9			High T_c due to stoich formation Depend. of T_c, resistivity & structure on chem. comp.studied
74	'77	Pollack IBM	3-1	?				Studies indicate a change of Nb-Ge bonds - from
75	'77	Gavaler	3-1/	films				The high T_c phase of Nb_3Ge films is stabilized by impurities O_2,N_2,SiH_4
76	'77	Schmidt, et al	3-1	sputter film				"X-ray diffraction patterns are given"
77	'77	Chu, Schmidt et al	3-1/A-15	Sputtered thin film				Resistive measurements - anomalies indicative of a phase trans. obs.@485°K in Nb_3Ge

Table 4 Properties of "Nb_3Ge"

Ref. No.	Year	Author	CMPD/ Stoich	How Prepared Film/Bulk/Size	T_c, °K	a_o A°	J_c A/cm^2	Additional Misc., etc.
78*	'77	Cox, Newkirk, Valencia	3-1/A-15	CVD Near Stoich				Order parameters were determined. Annealing converts to disorder (CVD 3-1) [illegible] Nb-rich A-15 [illegible] X-ray intensities measured
79	'77	Testardi	3-1/A-15	Thin films				Optimum values of T_c for Nb:Ge in the range of 2.6:1 - 3:1 up 95% single phase A-15
80	'77	Ghosh	3-1	Sputter ?	21.7			Several explanations given to explain/account for the T_c behavior
81	'77	Winter	Nb/A-15	Dispersed in a ductile Nb matrix				T_c & crit.mg.field strength. Properties increased by heating to 1500-2000, quench to 500°, anneal to 600-1000°c
83	'77	Webb	A-15			Disorder from radiation damage Highest cause of T_c depresion.		Highest T_c occurs at or near the stoich composition - concentration depends on T_c -13 & 2.6°K per at % excess Nb.
85*	'77	Newkirk, et al	3-1	Sputt & V-D film .63-20μm	21.5-20.0		1.6×10^6 @5KG $.85 \times 10^6$ 4.2°K	Temp dependence of J_c in 3-1 graphs in reduced coordinates $J_c(t) = J_c(0)\ H_{c2}(t)^n$
86	'77	Santhanam	3-1	CVD film/-12μm			3-1 5.145 5.3 a_o 10.148 c_o 5.152	Identified 5-3 by electron diffraction - calc. d spacings
87	'77	Gavaler	3-1	Sputtering high purity film	22			The higher T_c - possibly due to more crystal log. ordered structure. The film have T_c of >4°K higher than buk same stoich.
89	'77	Hull, Newkirk	3-1	CVD	21.09			X-ray powder diff as (t) 4.2 to ~675°K cell const. varied linearly from 150 ro 675°K. Expansion coef 6.09 x 10^{-6}/K. No evidence of a phase change.

Table 5 Properties of "Nb_3Ge"

Table 6 Properties of "Nb_3Ge"

117	'78	Braginski, Daniel Roland, etc.	3-1	CVD tapes 1 m long	20	(3-4) x 10^4 4.2°K tesla	H_{c2} = 29 tesla @ 4.2K low val. due to S-S phase
118	'78	Paidassi Fr.	3-1	CVD tapes	23.6 ± 0.2	10^5 18K	Broad transition (~5K)
119	'78	Gribillya USSR	A-15		25-30		Computer prog. to est. T_c 25-30K for A-15
124	'78	Dew-Hughes	A-15	Failed solid state diff. from ternary bronzes.			No Therm evidence that Nb_3Ge can be produced by solid state reaction dynamic
125	'78	Gavaler Braginski	3-1				O_2 conc. near substrate & large lattice parameters there - High-T_c Nb_3Ge - metastable phase from this region homo epipaxial
126	'78	Tsuei IBM	3-1	Amophous	3.9		Study of Sc & normal state properties of the amolph & crystal
129	'78	Zwicker Ger	3-1 A-15				H impurities lower T_c & J_c in A-15 Compounds
130	'78	Saur Ger	3-1	CO-Evap film 60-800nm		5.14	J_c & H_{c2}as f (T, thickness, orient.) No values given
131	'78	Chi; Schutz	3-1	dc sputter film			X-ray diff. - 3 broad peaks - no cryst. reflections
132	'78	Braginski	3-1	CVD 5-10m long tapes			H max = 20 tesla @ 4.2K
133	'78	Saur; Braun	3-1	dc sputter film .5 - 1.5μm	22.7	5.14	

Table 7 Properties of "Nb_3Ge"

During October and November the five "as cast" samples of compositions ranging from Nb 13.5% Ge to Nb 35.1% Ge were reduced to powders suitable for x-ray analysis. Sample holders were machined to various depths (.05, .1, .5 mm) to find the optimal depth with a minimum volume of sample. Current fluctuations which were probably due to gas in the old-x-ray tube, delayed the x-ray analysis of the samples until a substitute tube could be installed and tested. The preliminary x-ray analysis of the samples was completed by mid-December with no further problems in the instruments.

Sections of the diffraction patterns for the samples are shown in Figures 3 through 7 to help illustrate the results of the x-ray analysis.

The region from 34° to 44° is the most "active" of the entire scan for these Nb Ge samples. For that reason, almost all of the important information concerning the samples can be found in this 10° range. The locations of the peaks are the "fingerprints" of the sample, and each phase has its own distinct set of peaks.

The task of qualitative analysis, then, becomes that of isolating and identifying the "fingerprints" of each phase. The standards for identification are found in the x-ray powder diffraction files as shown in Figure 8. These files were first compiled and revised in 1957 by the American Society for Testing and Materials, and new sets are updated every few years as additional substances are indexed. Compounds are indexed according to their "d" spacing, that is, the distance between adjacent planes in the set (hkl) of the Miller indices for the crystal system. The "d" values can be determined from the 2θ peak location according to the condition for Bragg scattering,

$$d = \frac{\lambda}{2 \sin \theta}$$

where λ is the wavelength of the radiation. In this analysis, λ is the Copper $K\alpha_1$, radiation equal to 1.54051 Å.

The 35.1% Ge sample was extremely brittle. The entire sample (0.12 gm) was needed to fill the sample holder. Two separate scans were made in the course of the analysis, the second run being used for data collection. The peak locations indicate the following phases:

1) Nb_5Ge_3 (T) tetragonal

2) Nb_3Ge (c) cubic

This is in good agreement with what one would expect to find on the 35.1% Ge region of the Nb-Ge equilibrium phase diagram.

The lattice constant was calculated for the Nb_3Ge in this sample as $a_o = 5.165 \pm .002$ Å. Using the equation given by Matthias[4], the long range order parameter was determined to be S = 0.7. This implies that about a third of the germanium atoms are on the wrong site in the crystal lattice.

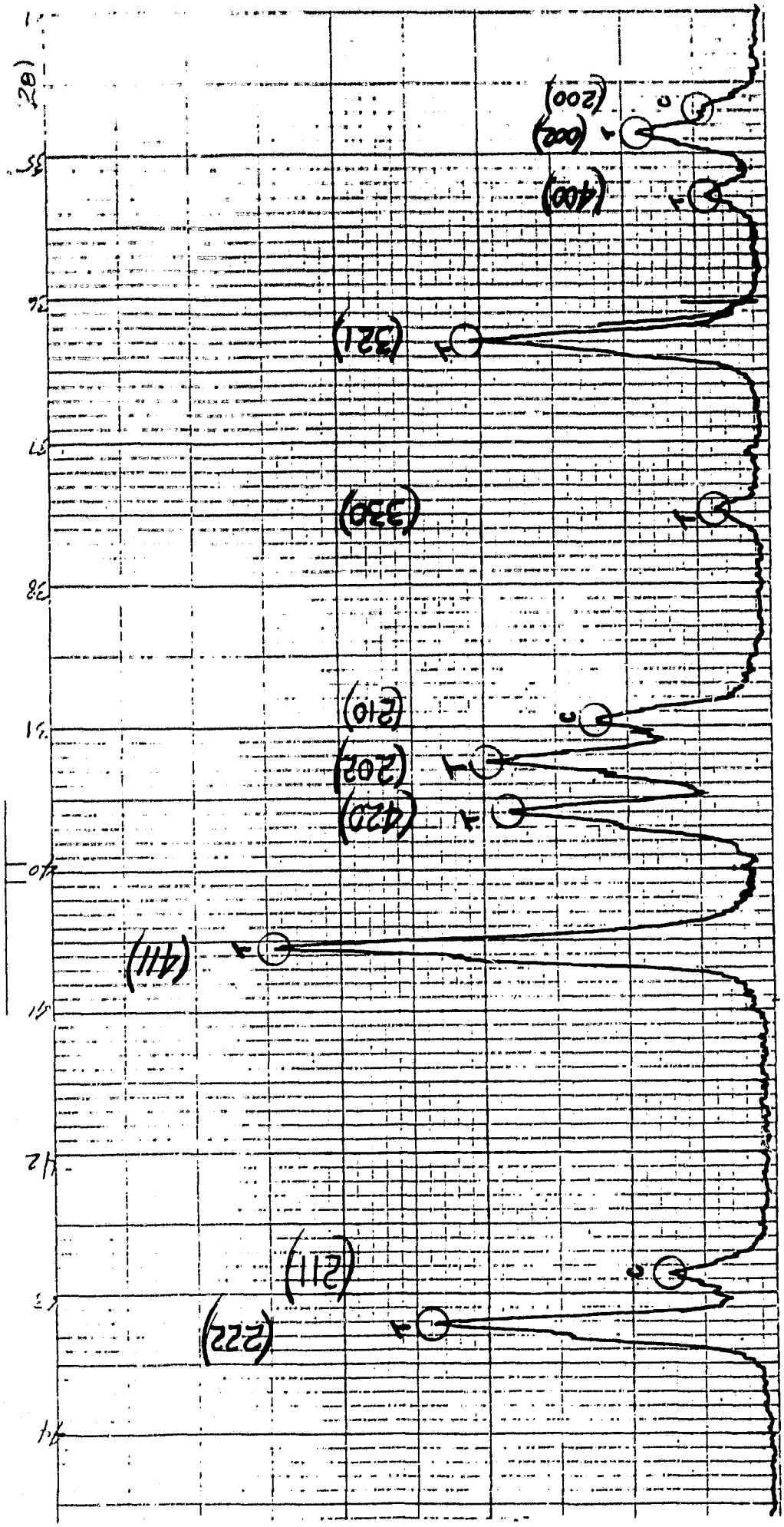

Figure 3: The Diffraction Pattern for the 35.1% Ge as cast sample indicates the following:

3 peaks of cubic Nb_3Ge with $a_o = 5.165 \pm .002$ Å

8 peaks of tetragonal Nb_5Ge_3

The Miller indices are listed above each peak.

Long Range Order Parameter for Nb_3Ge in this sample: S = 0.7

The peaks of the 27.1% Ge also line up with those of Nb_3Ge (C) and Nb_5Ge_3 (T) as found in files #10-296 and #8-354 respectively. The cubic lattice constant can be calculated for any peak of the set (hkl) according to the equation from lattice geometry,

$$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2}$$

In this sample $a_o = 5.167 \pm .002$ Å

Once again the x-ray analysis is in agreement with the phase diagram. Notice in Figure 4 that the Nb_3Ge peaks have greater peak heights than in the previous sample. This is because the composition is shifting toward the Nb_3Ge rich region, and the peak heights, or intensities, are proportional to the composition.

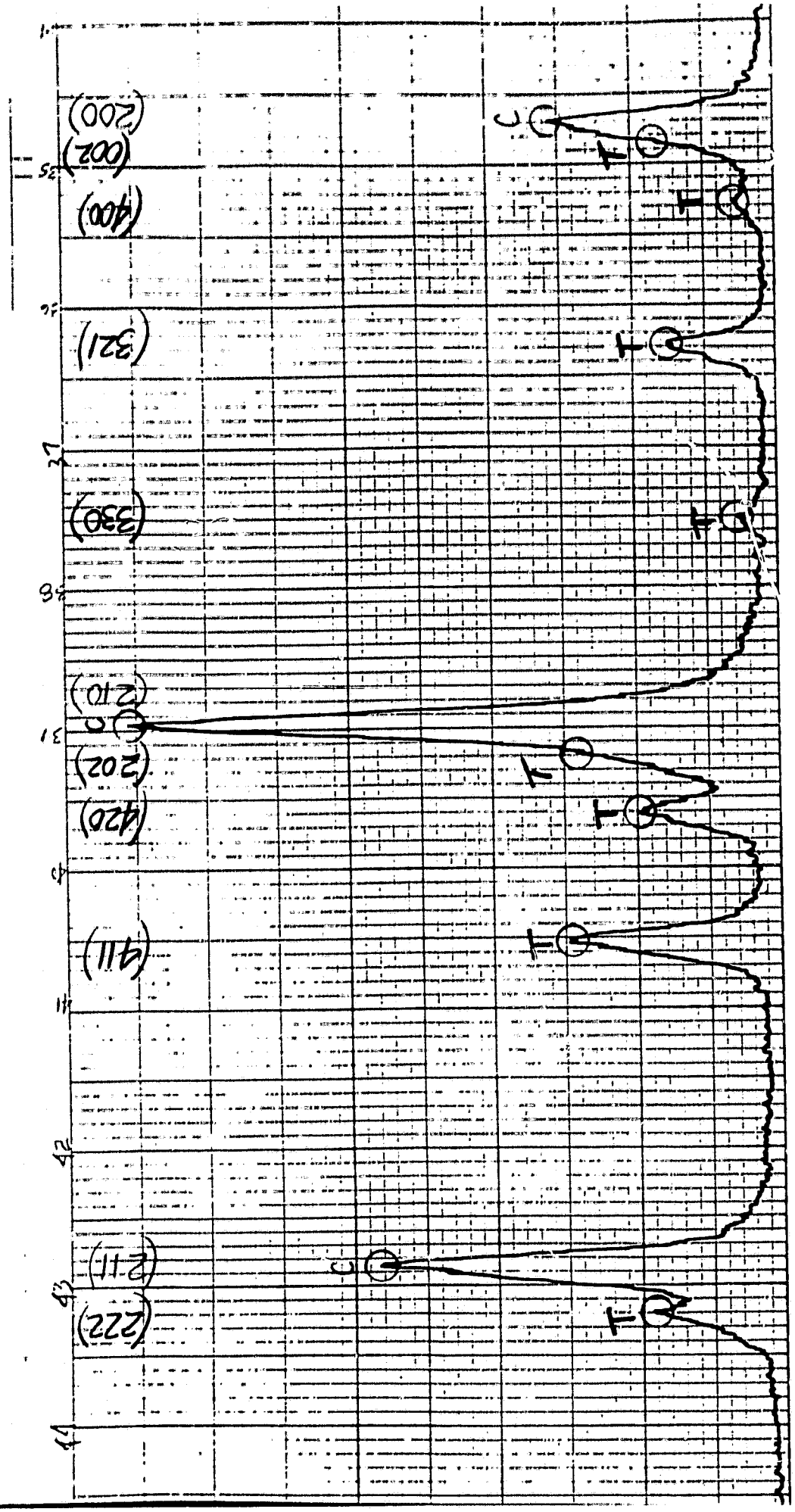

Figure 4: The Diffraction Pattern for the 27.1% Ge as cast sample indicates the following:

3 peaks of cubic Nb_3Ge with $a_o = 5.167 \pm .002$ Å
8 peaks of tetragonal Nb_5Ge_3

Long Range Order Parameter - indeterminate - out of range

The 22.1% Ge sample shows only slight traces of the Nb_5Ge_3 (T) phase in Figure 5. The bulk of the sample is the Nb_3Ge (C) phase. The lattice constant is $a_o = 5.166 \pm .002$ Å and the order parameter is S = 0.7.

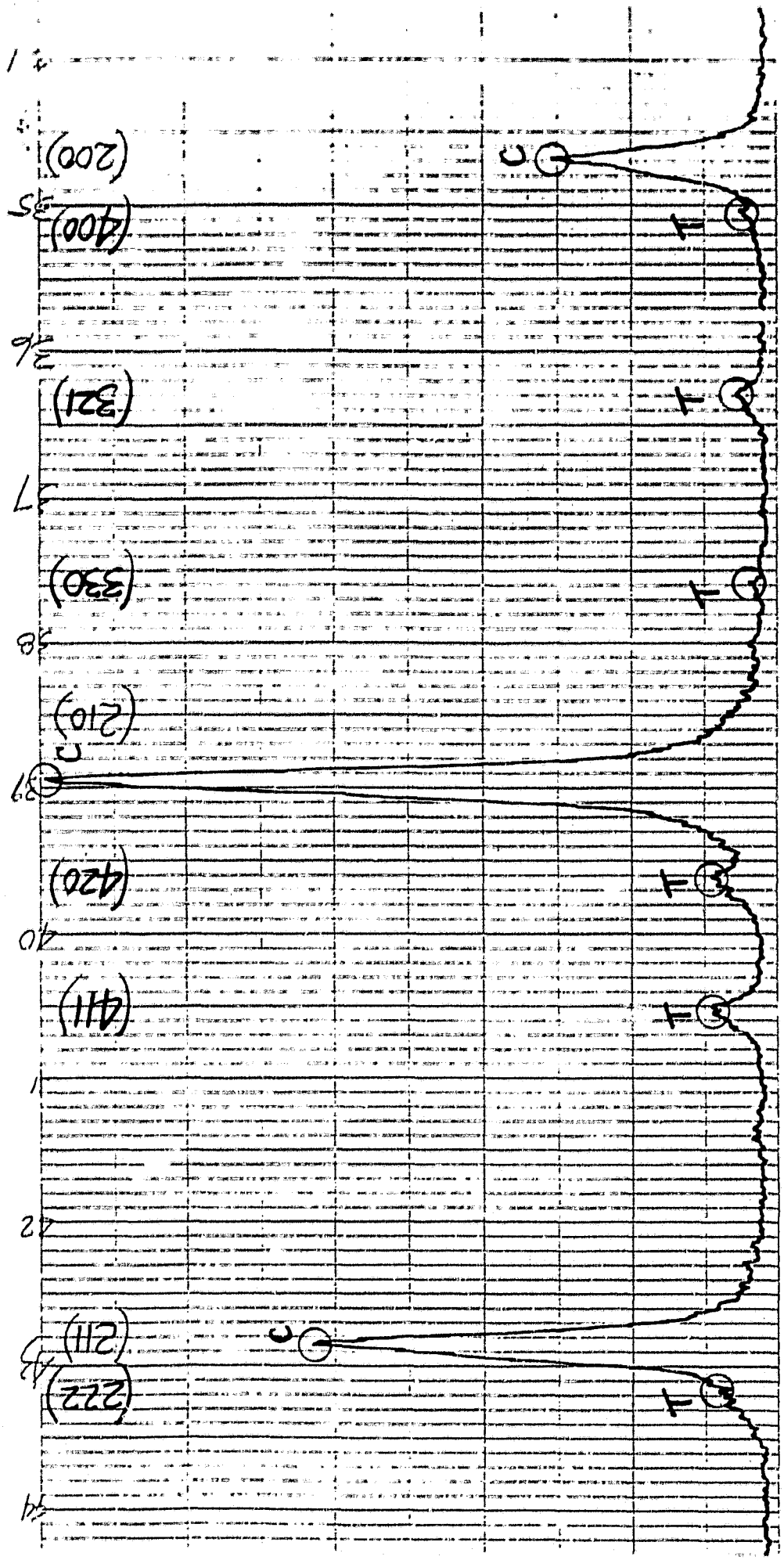

Figure 5: The Diffraction Pattern for the 22.1% Ge sample indicates:
3 peaks of cubic Nb_3Ge with $a_o = 5.166 \pm .002$ Å
6 peaks of tetragonal 5:3
Long Range Order Parameter for Nb_3Ge S = 0.7

On the phase diagram, the 18.2% Ge sample appears to fit in the middle of the β phase region. And at the first scan it was thought to contain only one phase also. However, investigation of the intensity ratios for the peaks revealed on additional phase, and another scan showed additional peaks, thereby verifying the presence of two phases:

Nb_3Ge (C) by file #10-296

Elemental Nb by file #16-1

The lattice constant for this sample is $a_o = 5.169 \pm .002$ Å.

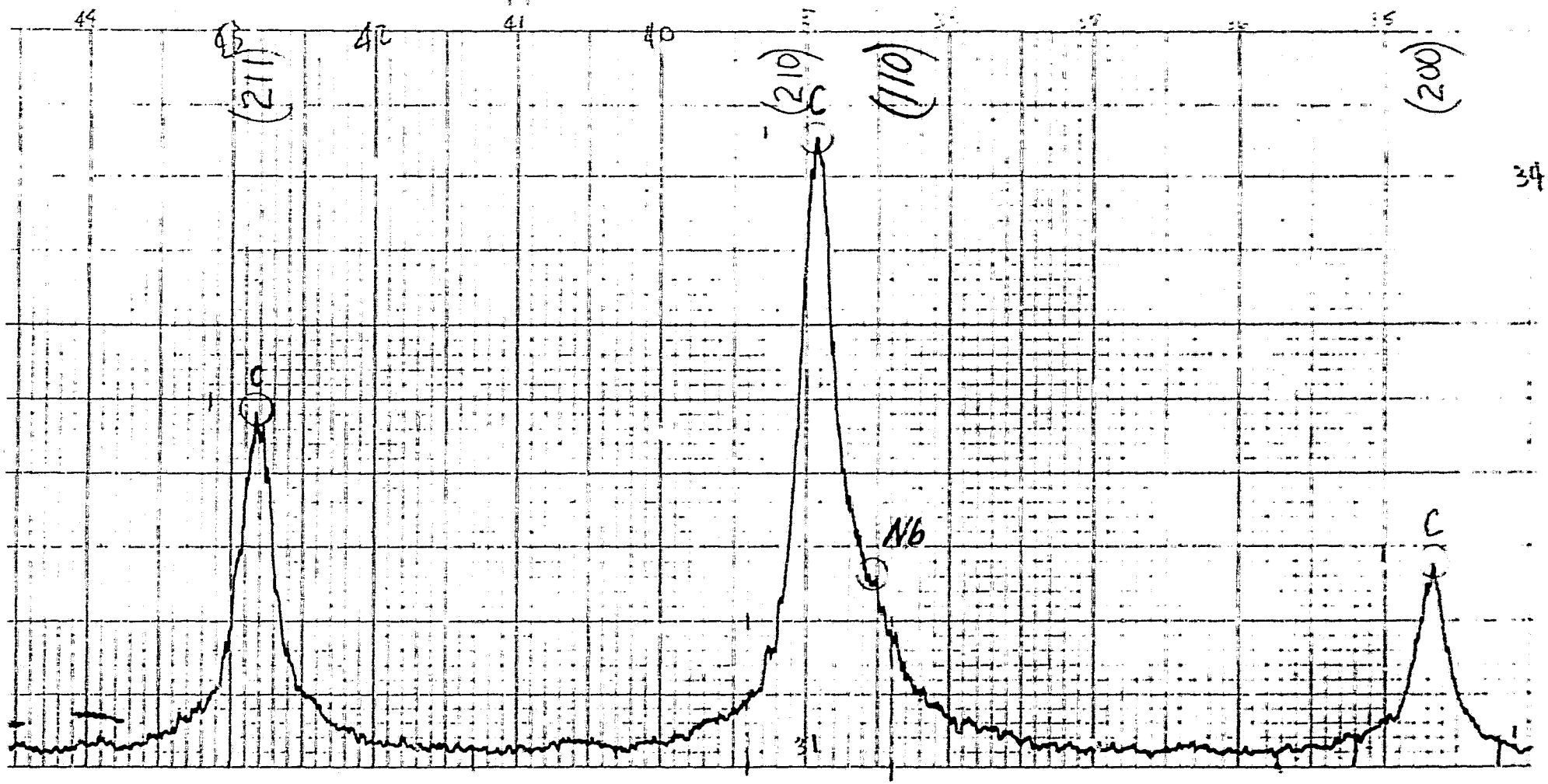

Figure 6: The Diffraction Pattern for the 18.2% Ge sample indicates 3 peaks of cubic Nb_3Ge with $a_o = 5.169 \pm .002$ Å

Long Range Order Parameter S = 1.85 – is out of range

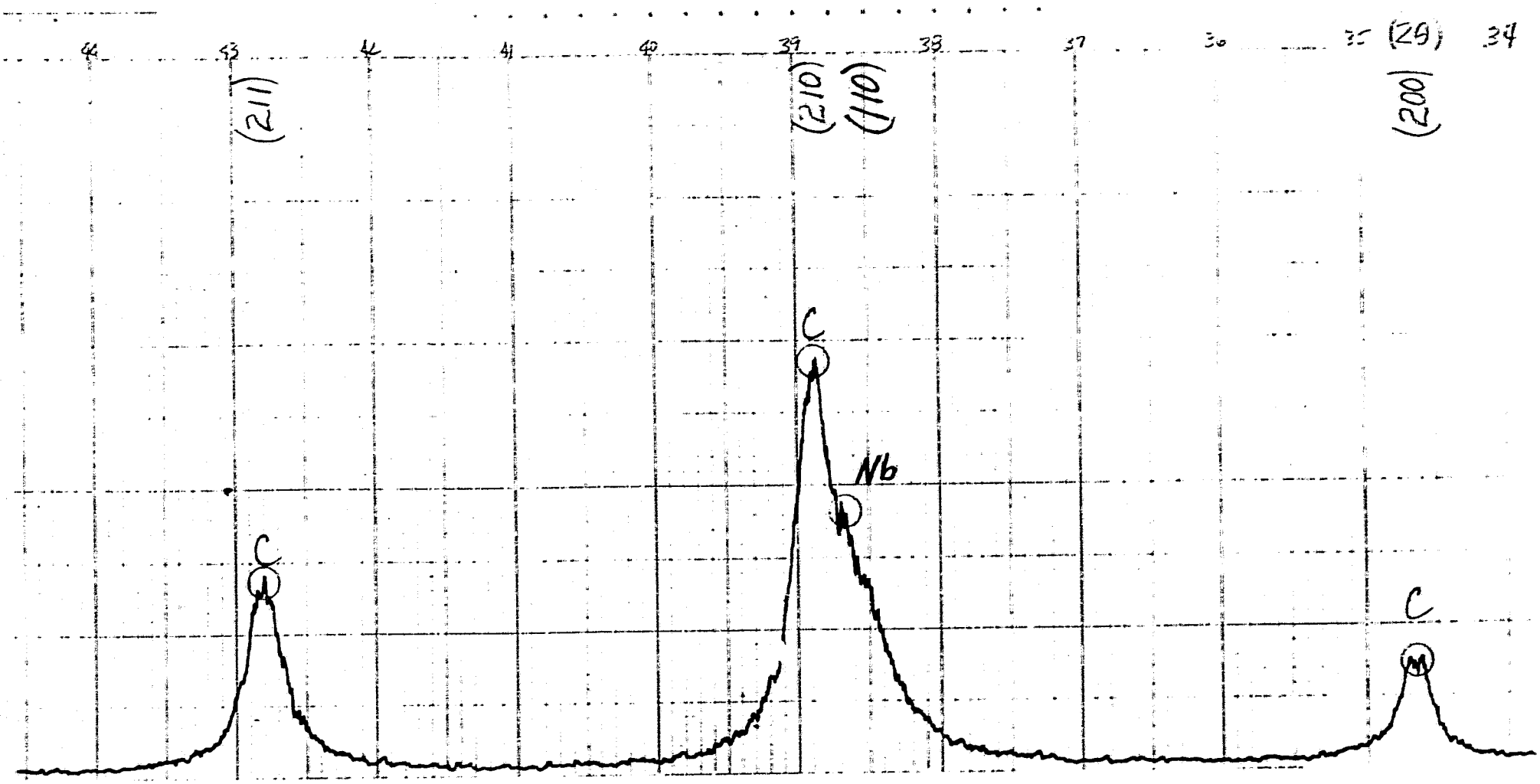

Figure 7: The Diffraction Pattern for the 14% Ge as cast sample indicates the following:

3 peaks of cubic Nb_3Ge with $a_o = 5.171 \pm .002$ Å.
1 peak of elemental Nb

Long Range Order Parameter is out of range due to over weighting of (210) peak from the Nb (110) peak.

'd' VALUES IN Å ±.002

[illegible]	[illegible] %	27.1%	22.1%	18.2%	14 [illegible]	Nb5Ge [illegible]	Nb5Ir3 Ti [illegible]	Nb5Ge3 Ti [illegible]	Nb
[illegible] 4%	[illegible] 8	2.583 11	2.584 21	2.585 24	2.586	2.563 50			
34.85	2.572 19	NR	NR .56 21				2.57 60	2.55 70	
35.29	2.541 8	2.542 [illegible]	NR				2.550 5	2.526 70	
[illegible]6.30	2.473 45	2.473 11	2.473 3				2.468 70	2.456 40	
37.5	2.397 [illegible]	2.396 [illegible]	2.391 <1				2.392 30	2.386 50	
38.70					2.325				2.33 100
38.76	2.309 25	2.313 10	2.311 100	2.312 100	2.315 100	2.293 100			
3[illegible]	2.[illegible] 47	NR	NR				2.305 80		
[illegible].00	2.279 43	2.271 13	2.273 6				2.271 80	2.273 80	
40.55	2.223 100	2.223 22	2.224 5				2.230 100	2.212 100	
4[illegible]	2.108 24	2.109 32	2.109 63	2.110 47	2.111	2.091 90			
43.[illegible]	[illegible].091 [illegible]	2.092 15	NR				2.087 50	2.076 80	

Table 8: The "d" values for the peaks shown in figures 3-7 are listed for comparison with the ASTM values.

NR = not resolvable

8-354 MINOR CORRECTION

d	2.23	2.31	2.27	2.58	$(Cr_3Nb_5)32J$ $(Ge_{0,84}Nb)$
I/I$_1$	100	80	80	60	GERMANIUM NIOBIUM

Rad. CuKα λ 1.5418 Filter Ni Dia.
Cut off I/I$_1$ VISUAL
Ref. NOWOTNY ET AL., J.PHYS.CHEM. 60 677 (1956)

Sys. TETRAGONAL S.G. I4/MCM (140)
a_o 10.148 b_o c_o 5.152 A C 0.508
α β γ Z 4 D_x
Ref. IBID.

ISOSTRUCTURAL WITH Si_3Mo_5.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
8.59	60	002			
2.550	5	400			
2.468	70	321			
2.398	30	330			
2.305	80	202			
2.271	80	420			
2.230	100	411			
2.087	50	222			
2.000	5	312,510			
1.806	5	402,440			

16-1

d	2.33	1.35	1.65	2.33	(Nb)32
I/I$_1$	100	30	20	100	NIOBIUM

Rad. MoKα$_1$ λ 0.709 Filter ZrO_2 Dia. 114.59mm
Cut off I/I$_1$ CALIBRATED STRIPS
Ref. HANAWALT ET AL., ANAL. CHEM. 10 475-512 (1938)

Sys. CUBIC S.G. Im3m (229)
a_o 3.3066 b_o c_o A C
α β γ Z 2 D_x
Ref. NADLER AND KEMPTER, ANALYT. CHEM. 31 1922 (1959)

εα nωβ εγ Sign
2V D 8.57 mp Color
Ref. C.C.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
2.333	100	110			
1.649	30	200			
1.348	30	211			
1.168	6	220			
1.045	10	310			
0.954	2	222			
.884	6	321			
.826	2	400			
.779	2	411,330			
.74	2	420			

10-296 MINOR CORRECTION

d	2.29	2.10	1.38	3.65	$(Nb_3Ge)8$
I/I$_1$	100	90	80	5	GERMANIUM NIOBIUM

Rad. CuKα λ 1.5415 Filter Ni Dia. 114.6mm
Cut off I/I$_1$ VISUAL INSPECTION
Ref. M.V. NEVITT, TRANS. AIME 212 350-5 (1958)

Sys. CUBIC S.G. $Pm3n$ (223)
a_o 5.168 b_o c_o A C
α β γ Z 2 D_x
Ref. IBID.

THE D'S ARE NOT CORRECTED FOR ABSORPTION EFFECTS.
THE PATTERN CONTAINED A SECOND PHASE WITH STRONG HIGH ANGLE LINES.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
(3.65)*	5	110	0.8610	60	600,442
2.563	50	200	.8493	60	610
2.293	100	210	.8380	70	611,532
2.097	90	211	(.817)*	2	620
1.801	10	220	(.808)*	2	621,540,443
(1.64)*	2	310	.7970	10	541
1.486	50	222	.7790	70	622
1.429	70	320			
1.377	80	321			
1.288	60	400			
1.233	5	411,330			
1.154	50	420			
1.126	60	421			
1.100	50	332			
(1.05)*	2	422			
(1.01)*	2	510,431			
0.9516	80	520,432			
.9426	70	521			
.9129	70	440			
(.888)*	2	530,433			

*CALCULATED BY J.V.S. FROM CELL DIMENSIONS.

Figure 8: Three X-ray diffraction file cards used for identification and verification of phases present in the samples studied.

26-684

d	2.21	2.46	2.27	2.55	$(Nb_3Ge_x)32U$
I/I1	100	90	80	70	Germanium Niobium

Rad CuKa λ 1.5418 Filter Ni Dia 114.6mm
Cut off SA I/I1 Visual Ideer.
Ref Private Communication, A. Storm, Bell Laboratories, Murray Hill, New Jersey, USA (1974)

Sys. Tetragonal S G I4/mcm (140)
a0 10.163 b0 c0 5.130 A C 0.5048
α β γ Z 4 Dx
Ref Ibid.

εα nωβ εγ Sign
2V D mp Color Grey
Ref. Ibid.

*Probable impurity line.
Sample arc melted from semiconductor grade Ge and 99.99% commercial Nb.
Superconductivity investigation by G. Hull showed ~2% superconducting phase indication Nb saturation in Nb_3Ge_x sample.
To replace 8-356

d A	I/I1	hkl	d A	I/I1	hkl
2.55	70	002	1.054	10	851
2.526	70	400	1.038	40	842
2.456	90	321	1.029	40	912
2.386	50	330	1.016	20	1000,860
2.273	80b	420	1.003	30	824
2.212	100	411	0.991	30	1011
2.076	60	222	.985	20	950
1.799	30	402	.976	50	833
1.768	50	521	.966	20	325
1.740	30	530	.9574	40	554,714
1.684	40	620	.9480	40	644
1.551	30	512	.9352	10	*
1.511	40b	541	.9219	10	952
1.464	50b	323	.9126	20	853
1.447	30	631	.9027	10	804
1.435	50	710,550	.8947	60	1121,1051
1.402	80b	413	.8892	60	824
1.383	10	*	.8793	10	*
1.362	30	622	.8760	50	644
1.343	10	721	.8704	20	1013

Figure 9: One of the revised and updated cards from the ASTM x-ray diffraction file.

The x-ray scan of the 14% Ge sample was just recently completed due to the fact that the 13.5% Ge sample studied earlier was found to be contaminated from the porcelain mortar set. The presence of Nb is very clearly seen in Figure 7, as well as the usual 3 peaks of Nb_3Ge The lattice constant is $a_o = 5.171 \pm .002$ Å.

During the next two months x-ray analysis efforts work will move from these "as cast" baseline samples to the very interesting samples solidified in the drop-tower. Higher accuracy A-15 lattice constants will emerge from slower scans and weighted averaging over the higher 2θ range of the diffractometer.

No problems are foreseen at this time which would impede this project.

The computer printout balance sheet shows for 31 December, 1979.

Encumbrance:	$1,937.60
Expenditure:	2,036.47
Balance:	6,189.96

The results of this preliminary analysis seem to be in good agreement with the expected results with respect to the equilibrium Nb-Ge phase diagram, and with the standard "d" values as found in the ASTM files.

Only two minor problems with the ASTM files have been noticed from this initial study. The first is due to a systematic error of $2\theta \cong .4°$ found in powder file card #10-296, which is the standard for Nb_3Ge. It is the low 2θ angle peaks that are in error, and the lattice constant calculcated from these peaks are not in agreement with the listed a_o value. In some cases the error was as great as -0.04Å. As a result of this finding, the confidence in the ASTM values has been questioned, and a search is being made for updated data which does not have the internal discrepancy of the #10-296 file card.

The second minor problem is due to the omission of several peaks in the updated file card #26-684. This card is meant to replace #8-354, the standard for Nb_5Ge_3. It is not known at this time why the peaks were left out, but an attempt is being made to contact ASTM for the reason. In this case it is the newer, updated data which is in question, contrary to what was expected.

Also the observation has been made the the optimal sample-holder depth is 0.1mm. This requires about 120 mg of solid alloy in order to grind enough powder to fill the sample holder. The x-ray scans can be made with less, but 80 mg is probably the very minimum weight necessary to analyze the sample.

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* - abbreviation for Superconductivity

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APPENDIX B

PROGRESS REPORT II

Contract NAS8-33548

August 1979 - February 1981

X-RAY DIFFRACTION ANALYSIS OF Nb_3Ge

by

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and

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February 1981

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OBJECTIVE

The objective here is to perform x-ray diffraction analysis on NbGe alloys quick solidified by supercooling in a free fall drop tower. Accurate lattice constant measurements yield the percentage composition of each phase while diffractometer intensity measurements yield the approximate percentage abundance of each phase and the long range order parameter.

In particular x-ray evidence is sought for the solidification of high superconducting transition temperature stochiometric metastable A-15 β -phase Nb_3Ge being formed by the large (~500°C) supercooling.

The equilibrium β-phase width is only 1% from 16 at. % G to 17 at.% Ge quite far from the stochiometric composition at 25 at. % Ge. The β-phase lattice constant should change from 5.167Å at 17 at. % Ge (equilibrium) to about 5.14 at 25 at. % Ge. Most of the two dozen samples reported herein were found to have β-phase lattice constants in the equilibrium range. One or two could be considered marginally in (i.e. in the fringes of) the metastable range with the desirable decreasing lattice constant.

X-Ray Diffraction of Nb_3Ge

A. Contract results have exceeded the proposed expectations in most areas.

1. Designed for only 9 months service - the service period of this contract has been doubled to about 18 months.

2. Lattice constant (β phase) accuracy ($\pm$ .002 Å) is an order of magnitude better than proposed.

3. An abondoned SSL Diffractometer has been refurbished with new transistorized components in the counting circuit. It is now in better than new condition serving the SSL Staff.

4. A rotation stage was borrowed from Army labs which allows high accuracy lattice constants measurements on the polished face of a sample without destructively powdering.

B. Results which thus far are less than expected.

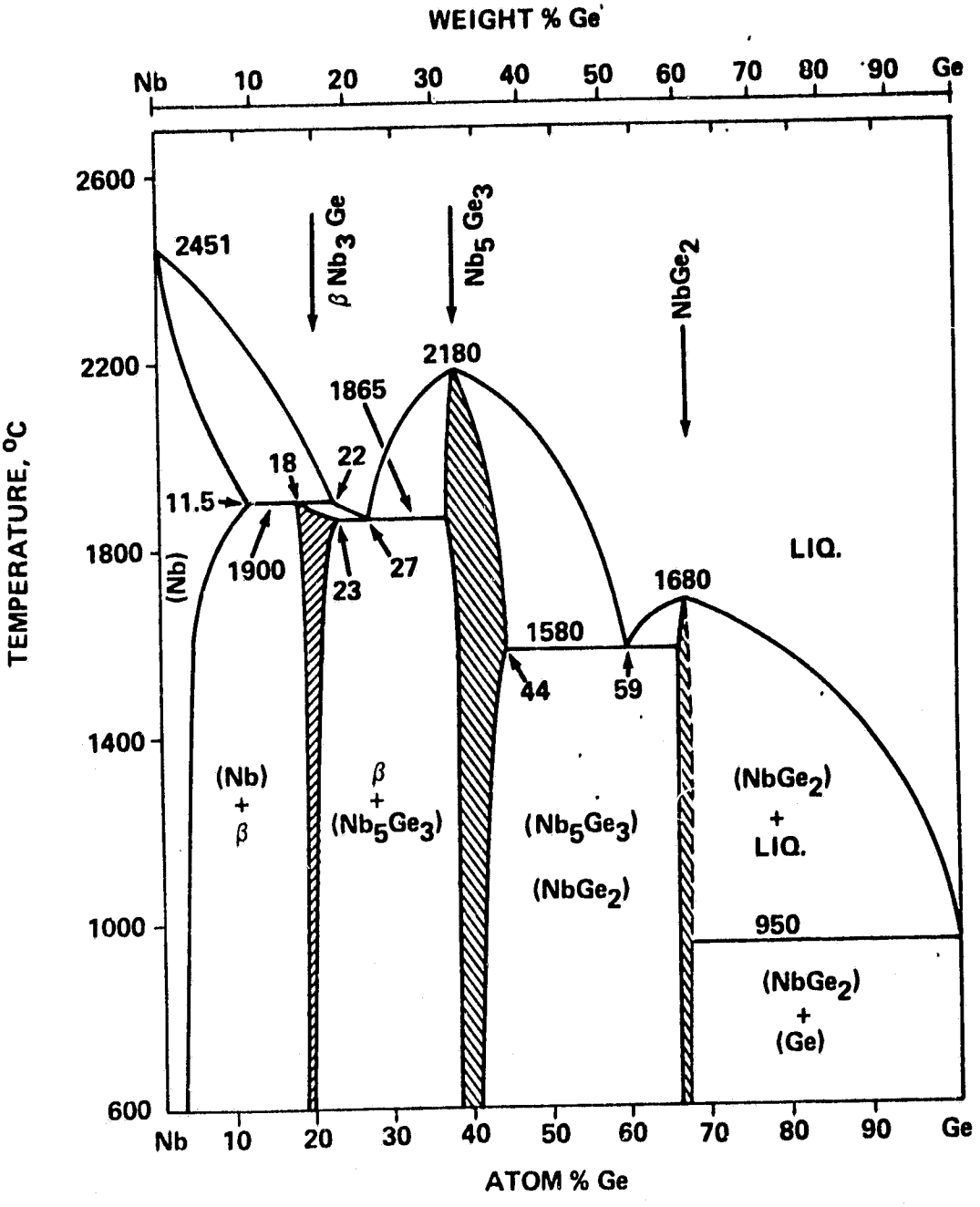
1. Calculated order parameters for the β phase (S) contained greater than expected error due to the presence of other phases (α and γ) in the samples analyzed. This will improve as the samples become more nearly single phase.

2. Evidence for the stochiometric β phase was not found; thus its volume fraction could be less than 20%. Past β stochiometric phases were surface located, however present ground polished and powdered samples are more volume related. Future powders should be ground from the surface of drop tower solidified samples to seek the most enrichment of the stochiometric (metastable) β phase.

Enhancement Techniques for Improving the X-Ray Diffraction of Nb_3Ge

1. Concentrate X-Ray analysis toward surface. Look for near stochiometric Nb_3Ge nearer the surface by using softer (larger λ x-rays) which are less penetrating.
2. Use near-grazing-incidence to reduce the x-ray penetration to provide a truer surface composition.
3. Generate more surface area by crushing into a thin film (using two cooled bricks) the undercooled drop. The sphere is diometrically opposite to the thin film in surface to volume ratio.
4. Add a cryowheel at the bottom of the drop tube to further hasten the quick freeze.
5. Run a second diffractometer recorder tracing to at $\frac{1}{2}°(2\theta)$ shift at 30 x and 20 x and 10 x attenuation to find the percentage at which the second phase becomes detectable.
6. Operate the 1024 channel multichannel analyzer in the subtract multiscaling mode with an Nb power to better electronically subtract the unwanted α-phase samples lines from the samples which are α β mixtures.
7. Strive both electronically (as above) and metalurgically for purer β samples which do not have the α and γ phases which by overlaping peaks alter the intensity rations needed to accurately calculate the long range order parameter.
8. Try for <u>more</u> O_2 in the drop tube - needed for forming the metastable phase.

From W. C. Mossatt, Ref. 8



Reference 1 (Cullity, 2nd Ed.) and Reference 2 (Klug and Alexander) provide excellent books for the reader interested in x-ray diffraction. Cullity provides a broad introduction for the introductory undergraduate with problem sets at the end of each chapter. X-Ray Diffraction Procedures for Polycrystalline and Amorphous Materials (by Klug and Alexander) provides an encyclopedia of practical up to date techniques. Examples of their style and applicability are given on the next few pages. This Report by no means represents the total contract output, as an interim (35 page) report was filed February, 1980. Also, about one third of our man hours were spent in NASA facilities as integral team members of the overall projects with data being passed to the C.O.R. on a daily basis.

In order to check the accuracy of the diffractometer, calibration scans were made using Standard Reference Material 640.

The powder was mounted in disk form in order to fit the sample holder.

Three sets of data were taken for dish #2. Each separate scan is given by the scan number.

Additional scans were made of these disks at random intervals in order to make a weekly check on the alignment. No data were calculated from these checks since they were basically routine in nature.

TABLE 1

CALIBRATION RESULTS

Sample	Scan #	Plane	N.B.S SRM 640 by Strip-Chart Recording	LATTICE PARAMETER (Å) by .05° Step-Scan	by .01° Step-Scan
Disk #1	1	(533)			5.43144
Disk #2	1	(620)		5.43142	5.43139
	2	(533)	5.43142	5.43141	5.43136
	3	(620)		5.43142	5.43138

From the three valves found by 0.01° step-scanning of dish # 2, the mean and standard deviation is (for N = 3) $X = 5.431377, \sigma = \pm .000015$ Å.

The predicted uncertainty is $\pm .0002$Å by eq (8-3).

Since the standard deviation is less than the theoretical prediction, then the mean value of 5.431377 Å is taken as the experimental value for the Si lattice constant.

The confidence in this mean value does not appear to be unwarranted. Comparing this mean value with the NBS value of the SRM 640 reveals:

$$\frac{a_{exp} - a_{NBS}}{a_{NBS}} \times 100\% = \frac{.0004970}{5.43088} \times 100\% = .00915\%$$

an accuracy of $\simeq$ 0.01% .

In order to make a check on the amount of experimental error introduced into the analysis, selected samples were x-rayed again. In this way the actual reproducibility could be compared with the predicted uncertainty.

All values are in Å units.

DT 085 ANNEALED

α_o	of (611) plane by 1/4° min strip chart recording	5.17574
α_o	of (611) plane by .01° step-counting with Quadratic Fit	5.17574

DT 097 ANEALED

α_o	of (611) plane by .05° step-counting with Quadratic Fit	5.16899
α_o	of (611) plane by .01° step-counting	5.16898

SRM 640

Dish # 2

α_o - (620) plane by .05° steps		5.43142
		5.43142
by .01° steps		5.43139
		5.43138
(533) plane by .05° steps		5.43141
by .01° steps		5.43136
by ¼°/min strip-chart		5.43142

All calculations were carried out to five decimal places in order to show that there is essentially no deviation in the values out to the fourth decimal place.

Assuming a 2θ uncertainty of 0.005°, the predicted uncertainty by equation (8-3) is ± .0002 Å. The mean and standard deviation for these samples is given below:

		$\bar{X}$		σ
DT 095 AN	(N = 2)	5.17574	±	0.00000
DT 097 AN	(N = 2)	5.168985	±	0.00001
SRM 640 Dish # 2	(N = 7)	5.43140	±	0.00013

In this analysis, the fact that the standard deviation is less than the theoretical prediction is a reliable confirmation of the precision, or reproducibility, obtainable from the x-ray diffractometer used for this study.

Comments:

1) All samples were prepared for x-ray analysis by polishing the solid to the interior, except for samples DT 081 and DT 094, which were ground to fine powders (325 mesh) before analysis. Also samples DT 212 and DT 224 were x-rayed on the exterior surface only with no polishing.

2) The silicon calibration dishes are made from NBS standard reference material 640 (silicon x-ray diffraction standard).

3) The overall compositions of the samples are approximate values based on the as-cast compositions.

4) The annealed lattice parameter values are those of the eight (8) samples which were annealed by Tom Rathz. The annealed samples were grouped into lots according to the annealing temperature. "A" group annealed at 550°C for 12 hours. "B" group annealed at 650°C for 12 hours.

5) If a phase was not found in the sample the lattice parameter is marked "N". If the phase was present, then the lattice constant value is listed in the appropriate column. If the phase was present in barely detectable amounts, then the lattice parameter could not be determined due to low signal to noise ratio. In this case the presence of the phase is qualitatively noted by a "Y".

6) The values for the lattice parameter were taken from the diffraction peaks at the highest 2θ angle possible. In the case of the α phase, the values were obtained from the (321) plane, $2\theta \simeq 133°$.

7) When the lattice value is given to 4 decimal places, the precision is $\pm$ .0002 Å; for 3 places, $\pm$ .002 Å.

8) λ = 1.54051 Å - Copper $K\alpha_1$ radiation.

TABLE 2

X-RAY DIFFRACTION ANALYSIS OF BULK "Nb_3Ge"

Sample	Overall Composition % Ge	LATTICE PARAMETERS (Å) α-phase	β-phase	γ-phase	ANNEALED LATTICE (Å) α-phase	β-phase	ANNEAL LOT (A=550°C) (B=650°C)
Cast # 1	13	3.2933	5.1763	N			
# 2	18	3.2908	5.1666	N			
# 3	22	Y	5.1667	Y			
# 4	27	N	5.1686	Y			
Cast # 5	35	N	5.168	Y			
DT 077	13	3.2911	5.1772	N	3.2939	5.1772	A
078	13	3.2911	5.1775	N	3.2948	5.1781	B
081	18	3.289	5.175	N			
082	18	3.2884	5.1767	N			
083	27	N	5.1683	Y	N	5.1684	A
085	18	3.2890	5.1756	N	3.2932	5.1756	B
086	18	3.2891	5.1756	N	3.2932	5.1756	A
090	27	N	5.1682	Y	N	5.1671	B
091	22	Y	5.1683	Y			
094	22	N	5.1664	N			
095	22	Y	5.168	Y			
097	22	3.2906	5.1677	Y	Y	5.1689	B
099	22	3.2901	5.1686	Y	3.2935	5.1698	A
212	18	3.2920	5.1773	N			
218	13	3.2866	5.1767	N			
220	13	3.2907	5.1749	N			
DT 224	22	Y	5.1725	Y			

A - 2 Phase

"ALPHA" Phase

Nb + Ge SOLID SOLUTION

The data for the lattice parameters of the different phases found in selected samples are presented in graphic form. The composition data for these samples were determined by EDAX by Dan Kinkle.

α-phase

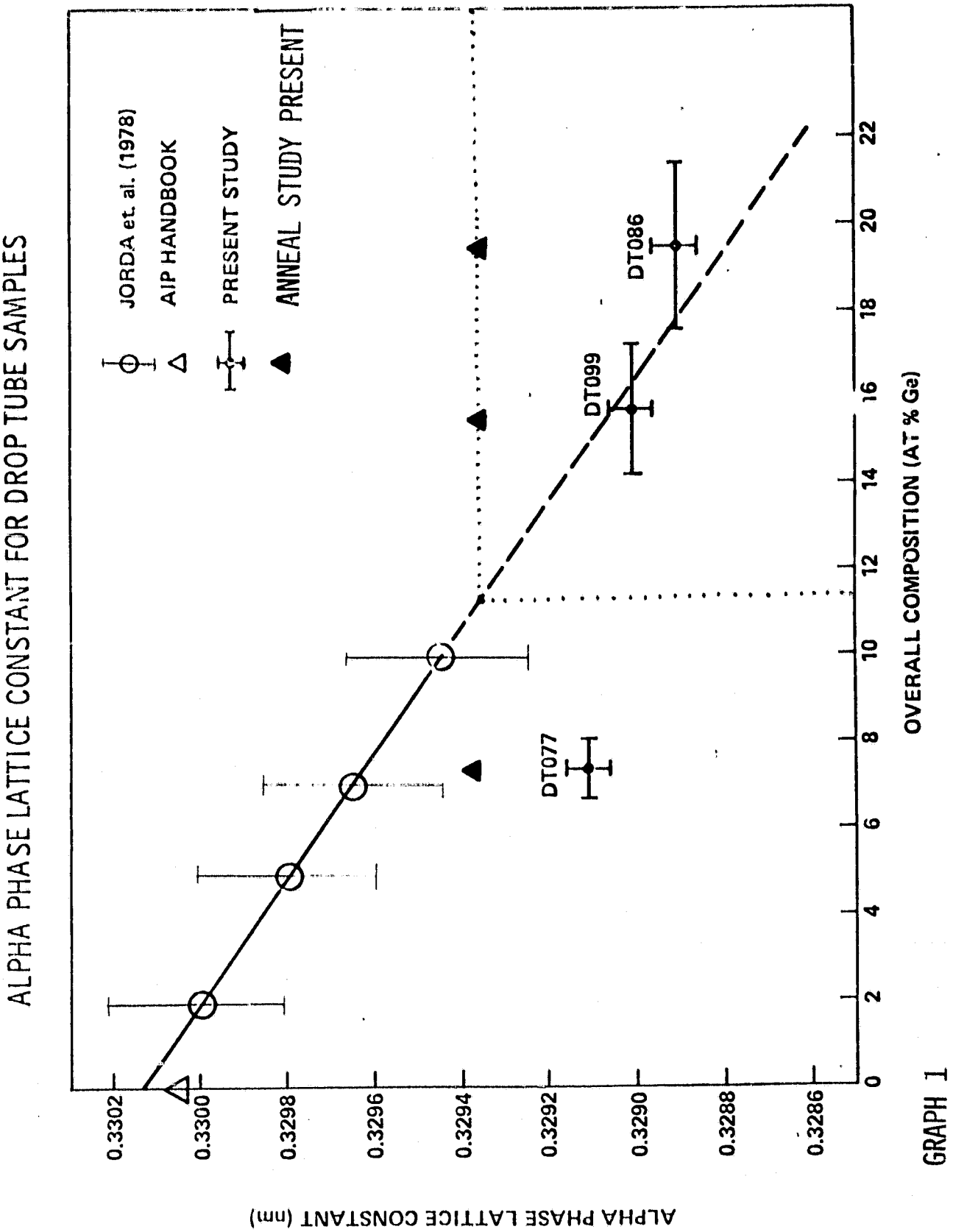
GRAPH I - Comp. vs. lattice parameters for DT 077, 086, 099 before and after annealing at 550°C for 12 hours.

Under equilibrium conditions, the maximum substitutional solution of Ge into the Nb lattice occurs at 1900°C. The overall composition of the sample is 11.5% Ge.[3]

The lattice constant corresponding to 11.5% Ge is (0.32934 nm) 3.2934Å. 3.2934Å is the equilibrium limit for the α-phase lattice constant according to these considerations.

Before annealing, these samples had α-phase lattice dimensions less than the equilibrium value. This fact implies the existence of a non-equilibrium α-phase.

After annealing, these samples had α-phase lattice constants equal to the equilibrium value. This implies that the annealing restored the non-equilibrium α-phase to an equilibrium condition.



GRAPH 1

% Change in α-Phase Lattice Constant

TABLE 3

$$\frac{a-a_o}{a_o} \times 100$$

a_o = 3.2934Å eq. limit at 11.5 % Ge Jorda (1978)

Comp % Ge	Cast Material	DT Sphere	Annealed Sphere	DT Flat	Annealed Flat	DT Splat	Annealed Splat
13	-.0015						
DT 077				-0.07	+0.02		
DT 078				-0.07	+0.04		
DT 218		-0.20					
DT 220st		-0.08st					
st = stinger attached							
18	-0.08						
DT 082				-0.15			
DT 085				-0.13	-0.004		
DT 086				-0.13	-0.004		
DT 212						-0.04	
22	Y						
DT 097		-0.08	Y				
DT 099		-0.10	+0.003				

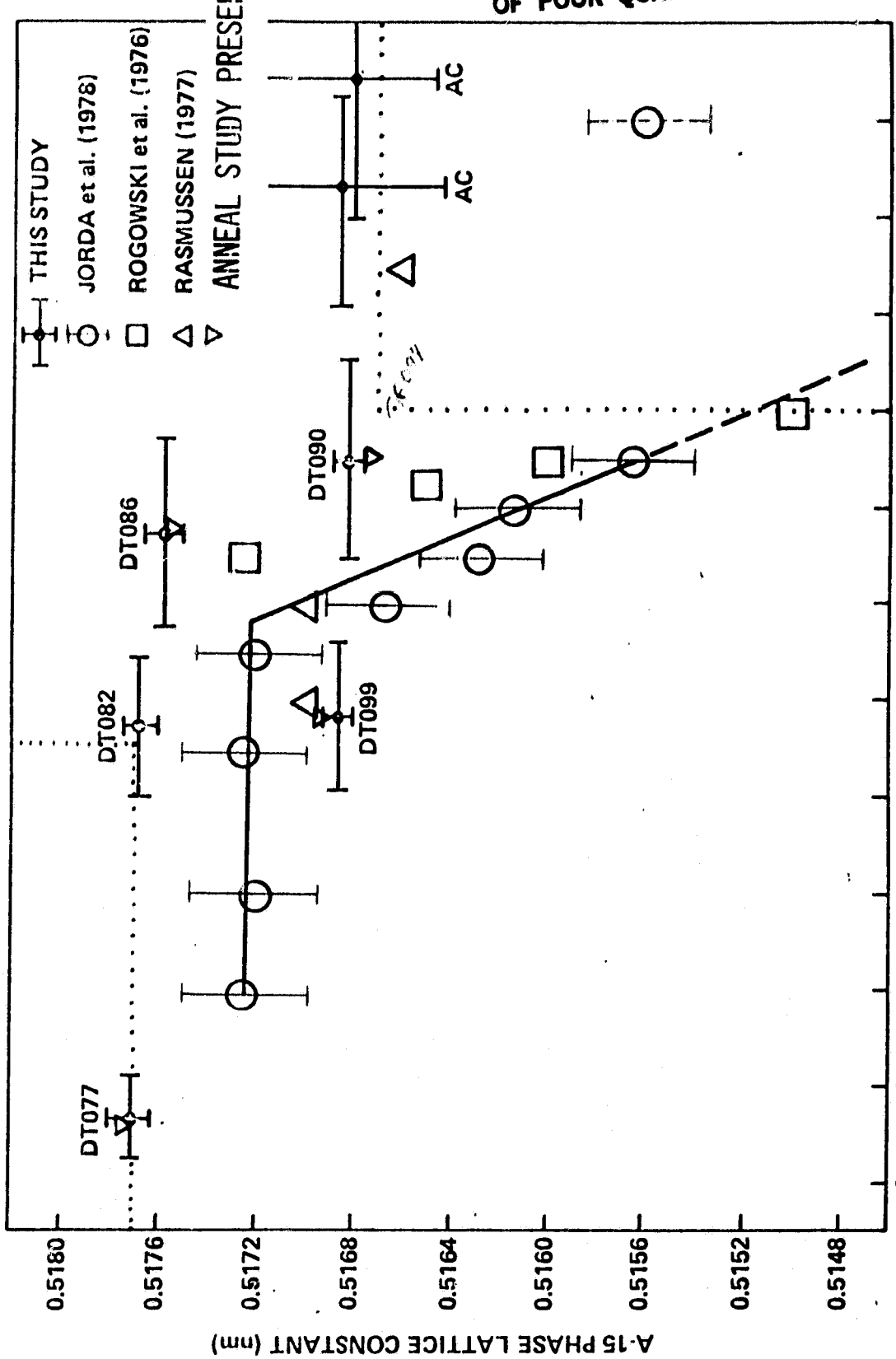
Any negative $100\ (a-a_o)/a_o$ is non-equilibrium α-phase. (The Nb + Ge solid solution.)

Annealing drives sample back to equilibrium value.

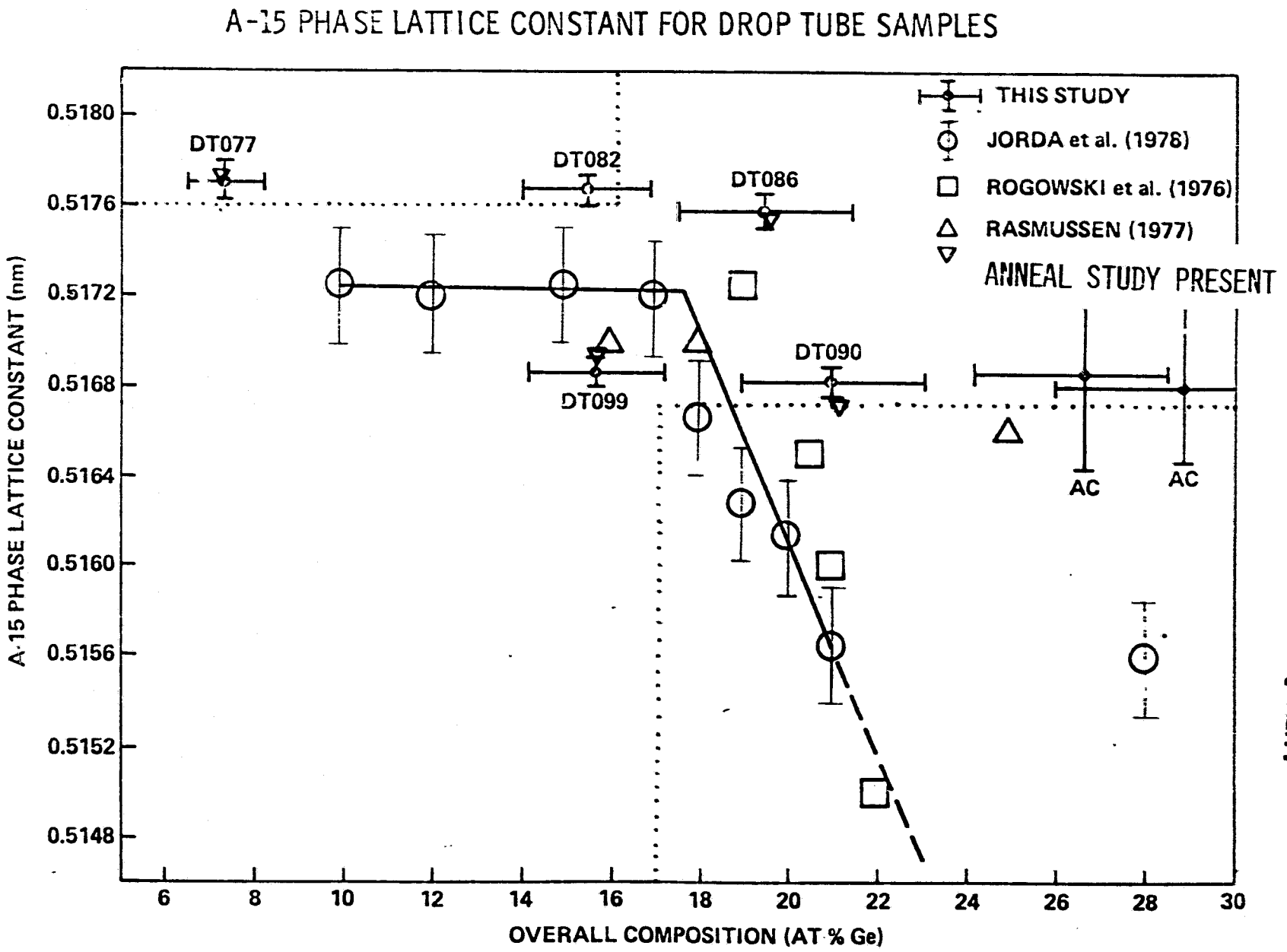
GRAPH II

Carpenters criteria for non-equilibrium is given by the dotted off area in the upper left and the lower right. Since the EDAX overall compositional error bars spread over a range of error of ~30% the lattice constants offer the only precise means of determination of whether or not a sample phase is stable. Note that overall composition in this study is the average over two or three pahses (α,β,γ) present in the sample and should not be confused with the narrow β-phase width from 16 to 17 at % Ge shown between the dotted lines.

Except for DT 086 and DT 099, the lattice constants seem to cluster about 5.176Å for the Nb rich end and 5.168Å for the Ge rich end of overall composition as expected. This study seeks to identify for the first time bulk Nb_3Ge samples with lattice constants in the range (17 to 22 at % Ge) as found by Rogowski[4] and Jorda[3] on thin films and splat cooled samples, as shown in Graph II.



GRAPH 2: CARPENTERS CRITERIA DOTTED INTO GRAPH



GRAPH 3: ROGOWSKI'S CRITERIA IN DOTTED LINES

A-15 Phase "Nb_3Ge"

Graphs 2 - 4

Overall composition versus A-15 lattice constant for selected samples before and after annealing. "AC" is as cost material.

Graph 2

The dotted lines mark the regions given by Carpenter (Ref. #5) for the limits of the Equilibrium A-15 Homogeneity range.

Graph 3

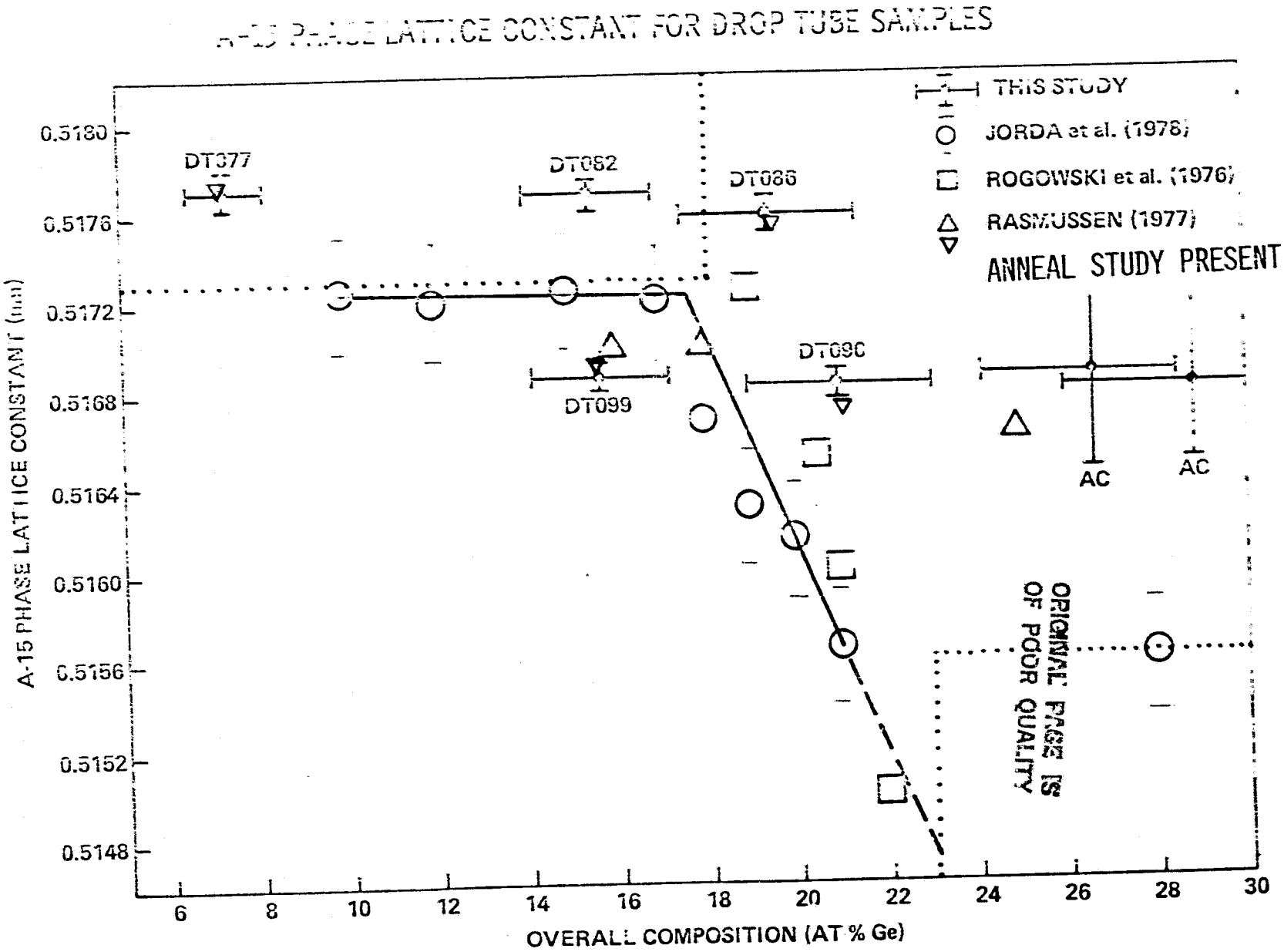
The dotted lines mark the regions given by Rogowski (Ref. #4) as the limits of the stable A-15 Homogeneity range.

Graph 4

The dotted lines mark the regions of the equilibrium A-15 phase range from data given by Jorda (Ref. #3) at the eutectic temperature (1865°C).

A sample must be within the dotted regions in order to be considered metastable according to the various criteria. This graphic form allows quick evaluation of a sample in terms of equilibrium,

It is almost possible for the A-15 phase in any given sample to be classified as either stable or non-stable, depending on whose criteria is used for the grouping. [i.e., Carpenter, Rogowski, Jorda.] In fact additional criteria are given by other researchers -Müller, Rasmussen, Newkirk, etc. It will require more time to put them all together.



GRAPH 4: JORDA'S CRITERIA IN DOTTED LINES

The ASTM Powder Diffraction File Card # 10-298 gives a value of 5.168 $\mathring{A}$ for the lattice parameter of the A-15 phase ("Nb_3Ge").

Using this value for a_o, then $\frac{\beta-\beta_o}{\beta_o} \times 100$ can be evaluated for each sample and evaluated with respect to the various criteria, where "β" is the lattice parameters for the A-15 phase in this case.

A-15 Phase

$\frac{\beta - \beta_o}{\beta_o}$ x 100 β_o - ASTM Value #10-298

TABLE 4 % Change in β-Phase Lattice Constant

Comp.	Cast Material	DT Sphere	Anneal	DT Flat	Anneal	DT Splat	Anneal	DT Stinger	Anneal
13	+0.16								
DT 077				+0.18	+0.18				
DT 078				+0.18	+0.20				
DT 218		+0.17							
DT 220st								+0.13	
18	-0.03								
DT 082				+0.17					
DT 085				+0.15	+0.15				
DT 086				+0.15	+0.15				
DT 212						+0.18			
22	-0.02								
DT 091st								+0.07	
DT 094						-0.03			
DT 095				0.00					
DT 097		-0.004	+0.02						
DT 099		+0.01	+0.04						
DT 224 (cup)						+0.09			
27	+0.01								
DT 083		+0.01	+0.01						
DT 090		+0.003	-0.02						

Jorda's Equilibrium Criteria

	Lattice
Nb-rich limit 18% Ge	5.173 Å
Ge-rich limit 23% Ge (at eutectic temp)	5.156 Å

According to Jorda's criteria:[3] (1) Nb-rich end - lattice constants greater than 5.173 Å are metastable. (2) Ge-rich end - The lattice constant must be greater than 5.156 Å in order for the sample to be metastable.

Rogowski's Equilibrium Criteria

	Lattice
Nb-rich limit 16% Ge	5.176 Å
Ge-rich 17% Ge	5.167 Å

According to Rogowski's criteria:[4] (1) The value of any $100(\beta-\beta_o)/\beta_o$ number less than -0.02 is still stable Nb_3Ge on the Ge-rich side. (2) Any positive number must be greater than +0.15 in order to be non-equilibrium A-15 (i.e. metastable) on the Nb-rich end of the homogeneity range.

Carpenter's Equilibrium Criteria

	Lattice
Nb-rich limit 15% Ge	5.177 Å
Ge-rich limit 22% Ge	5.167 Å

According to Carpenter's Criteria:[5] (1) Metastable Nb_3Ge on the Nb-rich end requires positive numbers greater than +0.17. (2) On the Ge-rich side, a negative number less in magnitude than -0.02 is still equilibrium.

A-15 Phase Conclusions

Considering the Nb-rich end it appears from all three criteria that several samples have the metastable A-15 phase. Annealing (650°C, 12 hours) appears to have no effect on the lattice constant in these samples.

The three criteria do not agree as well on the Ge-rich side of the A-15 phase. Carpenter and Rogowski are in close agreement. The recent work by Jorda of course has definitely different limits on this end of the equilibrium homogeneity range at the eutectic temperature.

By Carpenter and Rogowski criteria, it is possible that DT 094 formed a metastable A-15 phase during annealing.

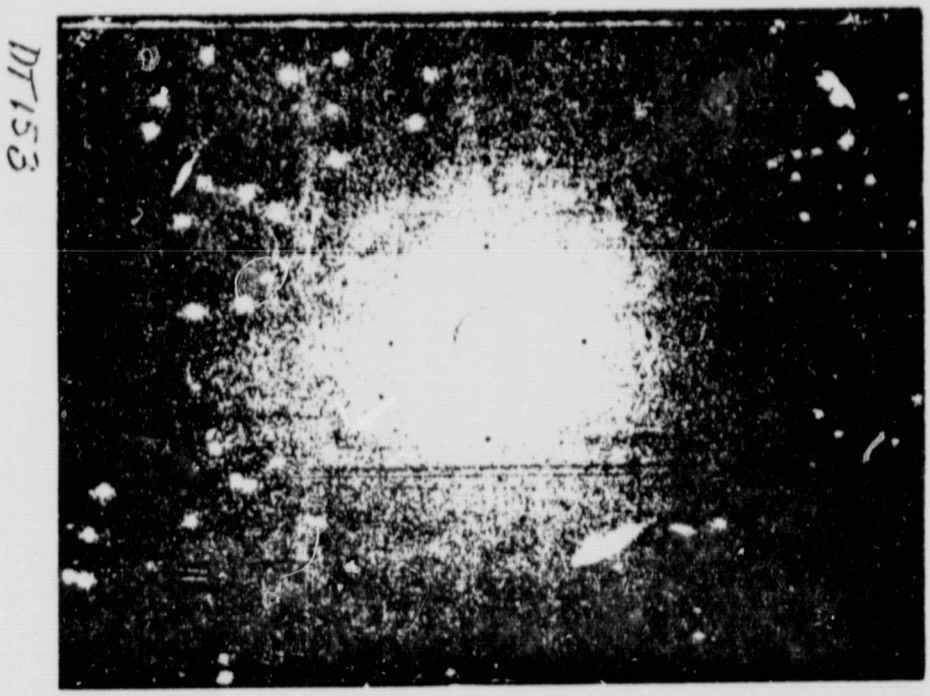
Likewise, under the same criteria, it may be that DT 090 formed a metastable phase during annealing.

According to Carpenter's Criteria:[5] (1) Metastable Nb_3Ge on the Nb-rich end requires positive $100(\beta-\beta_o)/\beta_o$ numbers greater than +0.17. (2) On the Ge-rich side, a $100(\beta-\beta_o)/\beta$ of -0.02 is still equilibrium.

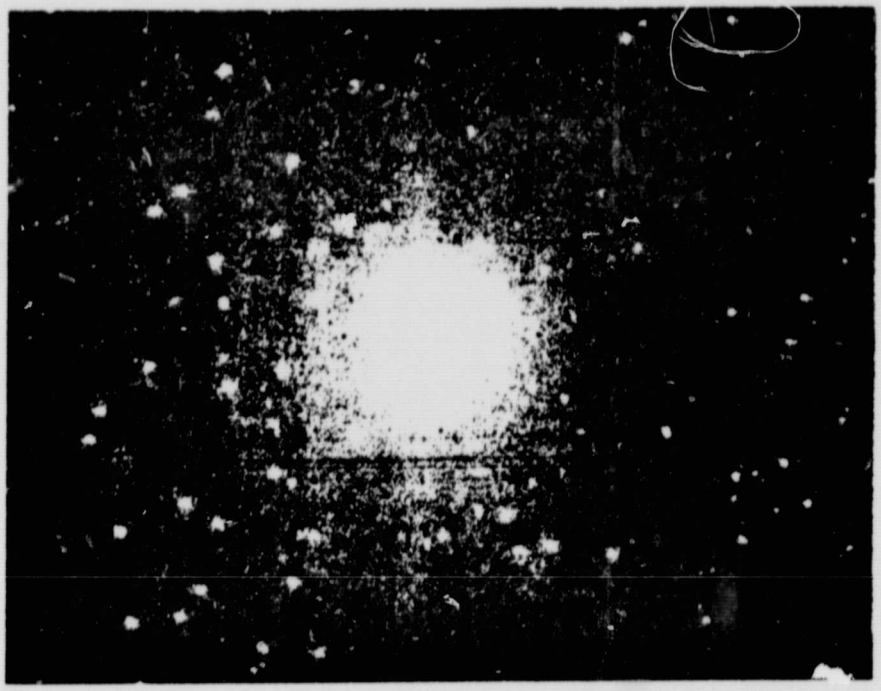
APPENDIX

Additional X-Ray Results

The effect of annealing on an Nb sphere processed in the drop tube is seen in the following back-reflection Laue photographs.



Sample DT153 before annealing. The presence of Debye rings through the elongated Laue spots indicates a deformed crystal.



Sample HDT153 after annealing at ~1400°C for 12 hours. The elongation and Debye rings are not present. With a polycrystalline specimen of randomly oriented grains a complete Debye ring is formed, because the normals to any particular set of planes (hkl) have all possible orientations in space; in a deformed single crystal, only fragments of Debye rings appear. We may imagine a circle on the film along which a Debye ring would form if a polycrystalline specimen were used, as indicated in Fig. 8-26. If a Laue spot then becomes enlarged as a result of lattice deformation and spreads over the potential Debye ring, then a short portion of a Debye ring will form.

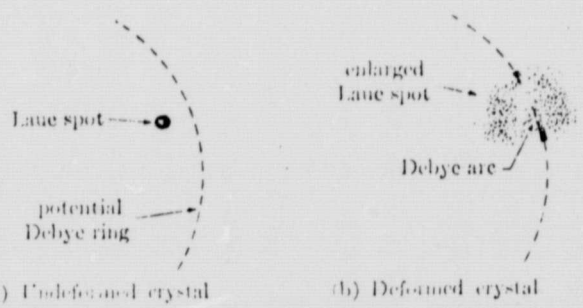


Fig. 8-26 Formation of Debye arcs on Laue patterns of deformed crystals.

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APPENDIX C

PROGRESS REPORT III

Contract NAS8-33548

March 1981 - June 1981

X-RAY DIFFRACTION ANALYSIS OF Nb_3Ge

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A revision of the previously reported "properties of Nb_3Ge" literature table was initiated to focus mainly on the studies of bulk forms of the alloy, including T_c and x-ray data.

The detector and electronic counting system for the x-ray diffractometer at S.S.L. were put into operation during this reporting period using the standard calibration, alignment, and operating procedures. The system is functional and adjusted for maximum efficiency.

Silicon powder calibration disks were used to check the accuracy of the x-ray system. The scan data for these disks yield a relative precision of 0.001%, and an absolute accuracy of 0.008%.

X-RAY DATA FOR ALLOYS

Sample DT 239 was polished and mounted for x-ray analysis, which revealed the following information:

1)	A-15 lattice parameter	5.1697 $\pm$ 2 Å
2)	Phases (relative volume per cent)	β (95) γ (5)
3)	Line width for (611) plane	0.30°
	corresponding lattice spread	$\pm$ 3mÅ

A slice from the Nb-25% Ge ingot was prepared for x-ray analysis which revealed:

1)	A-15 Lattice parameter	5.1689 $\pm$ 4 Å
2)	Phases (relative colume per cent)	β (64) γ (36)
3)	Line width for (611) plane	0.35°

A slice was also taken from the Nb-2% Ge ingot and x-rayed after polishing.

1) A-2 Lattice parameter — 3.2977 ± 2 Å
2) Phases — α (100)
3) Line width for (321) plane — 0.28°
   very noticeable broadening of peaks in this sample — probably indicative of compositional gradient.

Vegard's law for solid solutions can be used to predict the lattice parameter vs. solute concentration behavior. It does not hold, however, for the Nb-Ge solid solution (A-2 structure), designated as the 'α' phase, due to the dissimilar elemental structures of Nb (b.c.c.) and Ge (diamond cubic).

Since sample DT 224 was annealed before polishing, the x-ray data for DT 224 AN (annealed, polished flat) may or may not have any reliability when compared with previously reported data for DT 224 (unpolished, curved surface). In either case the analysis revealed for DT 224AN:

1) A-15 Lattice parameter — 5.1701 ± 4 Å
2) Phases — β (85)
   γ (15)
3) Line width (611) plane — 0.30°

An effort was made to introduce an internal calibration standard during the analysis of the Nb-Ge alloys. This was tried by mounting the alloy and Si standard powder 640 in the sample holder for simultaneous analysis. This does not appear to be feasible, however, due to the occurence of overlapping diffraction peaks for Si and the α (Nb-Ge solid solution) phase.

The line width of the diffraction peak (the (611) plane for β phase, (321) plane for α phase) is the width at half-maximum intensity.

Due to the flat shape of DT 243 (composition ~Nb-25% Ge) it was possible to x-ray this alloy in the "as processed" condition and obtain good S/N ratios.

1)	A-15 lattice parameter	5.1680 ± 4 Å
2)	Phases	β (91) γ (9)
3)	Line width, (b11) plane	0.30°

According to data given by Newkirk et al., [I.E.E.E. Trans., 1975 MAG 11(2) 221-4] on the variation of transition temperature with lattice spacing for Nb_3Ge, DT 243 would have a T_c of about 11 K.

An effort was also made to x-ray DT 244 in the bulk form without polishing, but the low S/N ratio was unsatisfactory for reliable analysis.

APPENDIX

The relative volume per cent of a phase present in an alloy can be predicted from the phase diagram for equilibrium conditions. Using the Lever rule one can obtain the following:

Volume percent of γ phase in Nb-Ge alloys

Composition	Temperature °C		
Nb- %Ge	600	1500	1865
22	12.5	6.3	0
25	38.5	30.7	16.7
27	63.6	54.5	40.0

The volume % γ phase can be measured experimentally and compared with these equilibrium values. The presence of a metastable phase in the alloy should be evident from this comparison providing the solidification temperature is known.

The relative volume % of the γ phase is found by normalization of the x-ray intensities measured for the (210) plane of the β phase and the (411) plane of the γ phase:

$$\frac{\gamma}{\beta + \gamma} \times 100\% = \text{r.v.\%}$$