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College of Engineering

DEPARTMENT OF INTERDISCIPLINARY STUDIES

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HOT TENSILE TESTS OF INCONEL 718

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Abstract

The physical metallurgy of near-solidus intergranular cracking in Inconel 718 welds was investigated. The data, although inconclusive, suggested at least two mechanisms which might explain intergranular cracking (microfissuring) in the heat-affected zone of several high temperature alloys. One theory is based on the separation of intergranular liquid while the other involves mechanical failure of solid ligaments surrounded by intergranular liquid. Both mechanisms concentrate strain in the grain boundaries resulting in low strain (<1%) intergranular brittleness. The mechanisms reported herein might also pertain to the physical metallurgy of casting, powder metallurgy sintering and hot isostatic pressing (HIP).

Introduction

The cracking and subsequent failure or rejection of weldments is the result of various causes. These causes are primarily dependent on the welding procedure and materials being welded. Two types of weldment failure in nickel-based alloys are solidification cracking and age cracking. Microfissuring is a solidification-type crack which forms in the heat-affected zone (HAZ) adjacent to the weld. These cracks are intergranular and often only one grain diameter in length.

The present methods for eliminating microfissuring are primarily procedural. Reduced welding speed and lower weld power both decrease microfissuring tendencies. A small grain size is also beneficial. The process of finding metallurgical remedies to the microfissuring problem is hampered due to a lack of understanding concerning the metallurgy of the cracking process. Specific points of contention concern the effect of chemistry, especially minor elements and impurities, the cracking process, and the physical metallurgy of the near solidus microstructures.

Studies by Yeniscavich (1966)¹, Owczarski, et. al. (1966)² and Weiss et. al. (1970)³ utilized hot ductility tests to provide information on a material's susceptibility to microfissuring. These investigations all reached different conclusions concerning the mechanism of microfissuring using this technique. A study by Savage et. al. (1976)⁴, which examined the HAZ microstructure, arrived at still a different conclusion concerning the mechanism of microfissuring. A complete literature review on the subjects of solidification

cracking, microfissuring, near solidus intergranular failure, and intergranular mechanical failure is presented in Appendix A. (This is an excerpt from progress report number #5). It is obvious from reviewing this literature that the microfissuring phenomena cannot be understood until the physical metallurgy of the near solidus material is better understood.

The present microfissuring studies stem from research initiated by A. C. Nunes⁵ at NASA/Huntsville (Marshall Space Flight Center (MSFC)). Dr. Nunes has calculated the temperature - strain response of the HAZ to a moving heat source. The heat source modeled the electron beam welding process so that the temperature - strain response of the HAZ could be represented by welding parameters. In order to predict when microfissuring would occur it was necessary to know the near solidus intergranular cracking behavior of the HAZ material. The cracking behavior had to be known in terms of temperature and strain so that it could be correlated with the welding parameters. The identification of the near solidus incipient cracking behavior of IN 718 was the objective of the present study.

It is necessary to investigate the initial cracking stage, or incipient cracking, of a material in order to investigate microfissuring. The fracture event is too far removed from the incipient cracking event to be of conclusive value for all but a few special cases. A program to determine near solidus incipient cracking in Inconel 718 was begun in June, 1979 under an ASEE/NASA summer Faculty Research

Fellowship (contract #NCT 01-008-021). The data generated by the above research necessitated further analysis and hence the present research program. The information reported herein builds on the previous research; however, this report is written to be a self-contained document. The reader does not need the previous final report to appreciate the current analysis.

Experimental Procedure and Results

The experimental plan was to plastically strain Inconel 718 at various temperatures near the material's solidus temperature. The plastic strain was varied between that needed to cause fracture and the strain needed to initiate a few small cracks. This was done at several temperatures so that the degree of cracking could be plotted against the plastic strain for each temperature of interest. This plot was used to extrapolate the degree of cracking to the incipient cracking strain. The incipient cracking strain thus obtained was plotted against temperature to give an incipient cracking envelope in strain-temperature coordinates. This plot can be used as a predictor of microfissuring when evaluated in conjunction with the Nunes analysis of HAZ strain and temperature.

Uniaxial tensile tests of Inconel 718 were used to evaluate cracking as functions of strain and temperature. The specimen dimensions were 0.635 cm x 2.54 cm x 60.96 cm. These rectangular bars were heated and strained in a vertical jacket furnace without a protective atmosphere. Table 1 gives information on the test temperature, maximum plastic strain, and whether or not failure occurred during straining.

A temperature profile along the specimen determined that a 7.62 cm zone of uniform maximum temperature existed along the specimen length. This zone was used for analysis of cracking tendencies at various maximum temperatures. Plastic strain was measured by a series of indentations which were measured before and after straining. One consequence of this strain measuring technique was to give strain at discrete locations as opposed to homogenous material strain. This becomes a source of discussion when analyzing the data. The measurement of strain marks was made with a machinist's microscope. Typical plots of plastic strain along the specimen length are given in Figure 1.

Cracking data was gathered by metallographically observing the centerline cross-section of the tensile specimen. By examining successive layers of the specimen cross-section it was determined that cracking originated at the centerline and that the centerline also contained the largest cracks. The centerline section of each specimen was polished and etched lightly to open the cracks and delineate the grain boundaries. All cracks were measured using a Leitz metallograph with micrometer eye piece. An arbitrary cracking parameter (N_c) was measured for each specimen by taking the length (L_c) times the width (W_c) of each crack and summing over all the cracks.

$$N_c = \sum_{i=1}^x (L_c W_c)_i \quad 1$$

(Note: cracks with a width smaller than 0.001 Filar units were too small to measure and arbitrarily given a value of 0.001.)

Typical plots of cracking along the specimen are shown in Figure 1. (Each crack number given in Figure 1 is the sum of all cracks over a 4 mm distance.) The extrapolation to the incipient cracking strain is shown for all specimens in Figure 2. The incipient cracking envelop as functions of temperature and strain is shown in Figure 3.

Discussion

The primary function of the present study was to quantify the incipient intergranular cracking behavior of Inconel 718. Figure 3 does this within the limitations of the experimental procedure. The greatest of these limitations was imposed by the strain measurement technique. The strain measurements were sensitive to local crack opening displacements as evidenced by Figure 1. The discreteness of the strain measurement marks proved a disadvantage since large crack opening displacements caused considerable scatter in the strain data. This is analogous to comparing the strain in the necked-down region of a tensile specimen to the strain in the unnecked region of the same specimen. This problem is manifested in the extrapolation to incipient cracking strain. A method is presently being considered which might enable the crack-opening displacement contribution to strain to be eliminated. The crack widths will also be normalized to a single value so that the data might be re-evaluated.

Due to the scatter in the strain data, the curve/curves of Figure 3 present some ambiguity in interpretation. Figure 3 may be taken to show a single, smooth c-curve indicative of a single cracking mechanism, or, it might be taken to show a double nose characteristic of two competing crack mechanisms. It seems appropriate now to discuss these possible interpretations of Figure 3 and provide supporting evidence.

Assume Figure 3 represents a single-nosed c-curve. Also consider Figure 4, which shows the plastic strain at failure as a function of temperature for Inconel 718. There are three distinct regions which

are labeled single intergranular crack growth, multiple intergranular crack growth and crack closure. The transition from crack closure to multiple crack growth appears to coincide with the initial loss of ductility as the test temperature increases. The transition from multiple crack growth to single crack growth appears to coincide with the complete loss of ductility. The apparent dependence of the grain boundary cracking mode on temperature could reflect microstructural changes over this temperature range. For instance, the fracture surface of specimens fractured at 2350°F gave an obvious indication of bulk solidus melting (Figure 5). The fracture surface at 2200°F also gave some indication that intergranular liquid was present at fracture (Figure 6). If this were interpreted similar to Yeniscavich, then the transition to single crack growth would correspond to complete grain boundary melting. The region of multiple crack growth, at temperatures between crack closure and single crack growth, would correspond to various degrees of partial grain boundary melting (Figure 7). In terms of Figure 3, there would be a single mechanism of cracking. The mechanism would be one of increased grain boundary wetting by intergranular liquid. The minimum in incipient cracking strain would correspond to complete grain boundary wetting by a very thin liquid layer. The temperature at which this thin liquid layer forms would be slightly below the bulk solidus of the material. The incipient cracking strain would actually increase as bulk-melting begins because the liquid is allowed to flow under strain. This flowing behavior was seen at fracture temperatures of 2350°F. Figure 8 shows a typical change in cross-section when this occurs.

Assume now that Figure 3 shows a double nosed set of c-curves. Two cracking mechanisms must be formulated to explain this situation. The most convenient mechanisms are a purely mechanical cracking mechanism for the low temperature α -curve and a grain boundary melting mechanism for the higher temperature c-curve. The author finds this situation more difficult to accept. The mechanical cracking mechanism would have to account for the change in mechanical cracking mode which occurs at the transition from intergranular crack closure to multiple intergranular cracking. The two mechanisms model would also have to account for the temperature of transition from mechanism one to mechanism two.

It is planned in the near future to examine the fracture surfaces of the specimens in the multiple-crack growth region. If indications of intergranular liquid could be found on these fracture surfaces it would support a single cracking mechanism, i.e., a single-nosed c-curve for Figure 3.

Conclusions

1. The strain measurement technique used in the present study is, by itself, inadequate for characterizing the plastic strain suffered during tensile testing. A better approach would be the present technique used in conjunction with techniques which average the strain over a larger gage section.
2. The near solidus intergranular cracking behavior of Inconel 718 exhibits several distinct phases. These are intergranular crack closure at approximately 120°C below the bulk solidus, multiple intergranular crack growth between 90°C and 40°C below the bulk solidus, single intergranular crack growth 40°C below the bulk solidus, and fluid flow at and above the bulk solidus.
3. The mechanism of HAZ microfissuring is not conclusively defined in the present program.
4. The incipient cracking strains presented in this paper should be reserved for Inconel 718 material with an ASTM grain size of #2 to #4.

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APPENDIX A
Literature Review

There are presently at least three mechanisms proposed to explain HAZ microfissuring and each would require a different method of metallurgical control. None of the three proposed mechanisms has conclusive experimental evidence either to support it or to disprove the others. The research program proposed herein would provide evidence of a conclusive nature on the role of intergranular liquid on HAZ microfissuring. The program results could then be used to identify solutions to the microfissuring problem.

4.0 Literature Review - HAZ Microfissuring

4.1 Hot Ductility Tests (Gleeble)

4.1.1 The hot ductility test is used to evaluate HAZ microcracking. It involves high strain rate fracture at temperatures up to the bulk solidus of the material. A thermal cycle is imposed on the metal to simulate the HAZ of a given welding process.^{4,5} The metal is fractured at a predetermined point in the thermal cycle to test the mechanical response of the HAZ microstructure. A typical result of such testing⁶ is given in Figure 2.

4.1.2 An area of controversy exists in explaining the rapid loss of ductility well below the bulk solidus temperature. Another area of debate exists in explaining the nil-ductility temperature. There is, however, good agreement that the nil-strength temperature corresponds to bulk intergranular melting at the solidus. There is also a good correlation between hot ductility data and sensitivity to HAZ microfissuring.^{6,7} Yet, there is no way of knowing what part of the hot ductility curve corresponds to HAZ microfissuring. Estimates^{1,8} of the maximum strain suffered by the HAZ are approximately 1%. This would indicate that the

incipient crack must form at or below about 1% strain. This value of strain does not correspond directly to hot ductility fracture strain because the fracture test is not necessarily an accurate indicator of incipient cracking. Tests on incipient cracking² under conditions approaching equilibrium do provide some insight on the relationship between hot ductility tests and microfissuring. These tests (Figure 3) indicate that at strains of 1%, incipient cracks do not form until the material approaches the nil-ductility temperature.

4.2 Theories of Nil-Ductility

4.21 Weiss, Grotke and Stickler⁹ studied the hot ductility response of Inconel 600. They suggested that the rapid heating of grain boundary precipitates suppressed diffusion, resulting in precipitate melting. They further suggested that the liquid immediately wet the boundary. The liquid film thickness was envisioned to increase with temperature from 20Å at the initial loss in ductility to 500Å at the nil-ductility temperature.

4.22 Veniseavish⁷ found good correlation between hot ductility behavior and HAZ microfissuring. He suggested that nil-ductility was initiated by low melting grain boundary films. These films were suggested to be eutectics formed by high concentrations of elements such as sulfur in the grain boundaries.^{8,10-12} This molten eutectic was believed to exist as low as 300°F below the bulk solidus of the metal. The intergranular melting was envisioned to begin at isolated positions thus causing initial loss of ductility. The ductility decreased with temperature as the liquid spread until nil-ductility resulted due to excessive grain boundary wetting.

4.23 Owczarski¹³ and Duvall and Owczarski⁶ suggested that a liquid intergranular film was not necessary for HAZ microfissuring. They suggested that the initial loss in ductility was caused by a change in deformation mode from transgranular to intergranular fracture.* They

*Whether or not the initial loss in ductility coincides with the onset of intergranular fracture is open to debate. The authors^{6,10} disagree on this point.

in turn suggested that HAZ microfissuring could occur by a solid state deformation mechanism. Although these authors⁶ observed intergranular precipitate melting in a number of alloys, they did not find that the melting necessarily corresponded to HAZ microfissuring.

4.24 A summary of the literature gives the following theories for HAZ microfissuring as interpreted from hot ductility tests:

1. Eutectic melting of the grain boundaries due to elements such as sulfur.
2. Liquid grain boundary films due to the melting of intergranular precipitates.
3. Solid-state deformation concentrated in the grain boundaries such as grain boundary sliding.

Note: More recent works by Savage and Nippes with Miller,¹⁴ Szekeries¹⁵ and Goodwin¹⁶ have also addressed HAZ microfissuring. These studies involved constitutional liquation of sulfide inclusions and unidentified spherical inclusions, eutectic melting of grain boundaries, grain boundary liquid distribution, and the effect of liquid-solid surface tension on grain boundary wetting. None of this work, however, was done in the spirit of the hot ductility test upon which the present proposal is based. It is hoped that the proposed research will contribute to the interpretation of the above studies and help relate their results to previous and future works.

4.3 Hot Tensile Test

4.31 This author^{2,3} evaluated the incipient intergranular cracking behavior of Inconel 718. The study was designed to determine the

incipient cracking behavior as a function of both total plastic strain and temperature. During these tests, a fracture curve (Figures 2 and 3) was generated similar to the hot ductility curve. Unlike the hot ductility test where an exacting thermal cycle is imposed on the metal, the hot tensile test allowed the metal to approach equilibrium. This was done by a slow heat-up followed by holding the specimen at a predetermined temperature for approximately five minutes before fracture. It is apparent that Inconel 718 exhibits all the characteristics of the hot ductility test even when given a long heat-up and soak time at the fracture temperature. This suggests that the rapid thermal cycling, as used in the hot ductility test, may not be as critical to evaluating HAZ microfissuring as currently believed.

5.0 Literature Review - Analysis of Theories

5.1 If HAZ microfissuring occurs due to partial or complete melting of grain boundaries regions, then it can be considered as a type of solidification cracking. Several of the theories associated with solidification cracking in welds and castings are helpful in interpreting HAZ microfissuring due to grain boundary liquation. A primary consideration in theories of solidification cracking is the distribution of the liquid phase.¹⁷⁻¹⁹ Since these theories concern deformation above the bulk solidus of the metal, it would be imprudent to extend them directly to HAZ microfissuring which is thought to occur below the bulk solidus. However, the following analogies seem appropriate and of some value in understanding hot grain boundary failure near the solidus. Bear in mind that theories of sub-solidus and super-solidus cracking differ primarily in the volume of liquid present in the grain boundaries.

5.2 Yeniscavish^{7,10} postulated that liquid was partitioned on the grain boundaries and gradually wet the boundary as the temperature approached the bulk solidus. Much work^{19,25} has been done on the distribution of intergranular liquid pertaining to solidification cracking. Smith²⁰ showed that interfacial energies should control the measurable dihedral angle (θ) according to:

$$\gamma_{SL}/\gamma_b = k_1 \cos \theta/2$$

where γ_{SL} is the solid-liquid surface tension and γ_b is the grain boundary surface tension. Smith and Williams²¹ showed that the distribution of liquid on the grain faces was dependent on γ_{SL}/γ_b and correlatable to θ . The dihedral angle has been shown to correlate with cracking²⁴ and be a function of temperature,^{23,25-27} stress,^{27,28} and trace elements.²³ The studies of liquid distribution lend credence to Yeniscavish's model of increased intergranular wetting by a liquid phase with increasing temperature. However, experimental evidence to support the eutectic melting of grain boundary regions is open to question. This theory could be disproved if it could be shown that initial bulk solidus melting, at the grain boundary, did not completely wet the boundary.

5.3 Weiss, et al.⁹ postulated that constitutional liquation of intergranular precipitates resulted in immediate wetting of the grain boundary. The resulting grain boundary film was expected to increase in volume with increasing temperature. Several studies²⁹⁻³² of solidification cracking have involved film stage analysis. Saveiko²⁹ postulated

that the thickness of the film was directly related to the film strength in a manner similar to Weiss et al.⁹ Experimental evidence^{6,9,13} supports intergranular precipitate melting in the HAZ. However, there is not conclusive experimental evidence to support complete wetting of the grain boundaries by initial precipitate melting. Nor is there any evidence to explain why this film thickness should grow with increasing temperature below the bulk solidus. Dihedral angle measurements should differentiate if melted precipitates wet the boundary or remain isolated.

5.4 Owczarski¹³ has suggested that HAZ microfissuring results from solid-state deformation initiated by the transition from transgranular to intergranular failure. This is suggestive of the equicohesive temperature developed by Jeffries.³³ The mechanistic cause of the equicohesive temperature is ill-defined. It appears that studies involving hot creep rupture offer better models from which to evaluate HAZ microcracking due to solid-state deformation. Ashby^{34,35} has developed a method of expressing deformation processes, accommodation processes, and typical engineering tests on a deformation map as a function of stress, strain rate, grain size, and temperature. The hot ductility test would fall in a region of power-law creep controlled by lattice diffusion. Other active mechanisms would be grain boundary sliding and dynamic recrystallization. The deformation maps presented by Ashby are not all inclusive. Consideration must also be given to grain boundary sliding mechanisms described by Gifkins³⁶ and grain boundary dislocation mechanisms discussed by Hirth³⁷.

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5.5 If it were possible to eliminate intergranular liquid theories of HAZ microfissuring by dihedral angle studies, then attention would be directed toward determining the active deformation mode in hot ductility tests. However, because of the evidence for intergranular precipitate melting, the intergranular liquid theories should be investigated first.

6.0 A New Theory of HAZ Microfissuring

6.1 None of the present theories for HAZ microcracking give an acceptable description of hot ductility behavior just below the bulk solidus. These theories do have their strong points and by combining them a new theory can be presented.

6.2 Experimental evidence shows that intergranular precipitates melt in the HAZ. It is postulated that the initial melting of these intergranular precipitates corresponds to the initial decrease in ductility during the hot ductility test. The initial melting is characterized by a large dihedral angle ($\theta \geq 90^\circ$). As the temperature of the test is increased, the dihedral angle of the melted precipitates increases as shown for other systems ($90^\circ > \theta > 0^\circ$).^{26,27} This results in a gradual loss of ductility as the temperature increases. The nil-ductility temperature is characterized by complete or near complete grain boundary wetting ($\theta \approx 0$) (Figure 4).

6.3 The proposed model could help explain several characteristics of the hot ductility test and HAZ microfissuring. The various susceptibilities of different alloys to HAZ microfissuring could be explained by the influence of bulk and/or precipitate chemistry on the balance of surface tensions. (The balance of surface tensions controls the

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APPENDIX B

Tables

TABLE 1
Hot Tensile Data

Specimen	Test Temperature	Maximum Strain (% ϵ_p)	Number of Cracks		
			Large	Medium	Small
7 ^{4,5}	2000	40 %			non-fracture
6 ^{1,4}	2000	10 %			non-fracture
8 ³	2100	45 %			non-fracture
1 ^{1,4}	2100	17 %			non-fracture
20	2100	5 %			non-fracture
22	2100	1.4%			non-fracture
13	2150	26 %			Fracture
18	2150	4.5%			non-fracture
19	2150	1.9%			non-fracture
14	2175	11 %			Fracture
16	2175	5.6%			non-fracture
17	2175	4 %			non-fracture
21	2175	1.7%			non-fracture
4 ¹	2200	0 %			Fracture
9	2200	3 %			Fracture
15	2200	0.7%			Fracture
10	2300	0 %			Fracture
2 ¹	2350	4.5%			Fracture
11	2350	3 %			Fracture
12 ²	2350	?			Fracture

1. cross head displacement rate - 2 inches/minute
2. fracture occurred 2 inches from nearest strain mark
3. small cracks found inside grains resulting from grain boundary migration
4. metallography for cracks not performed due to difference in strain rate
5. cross head displacement rate - 0.5 inches/minute

APPENDIX C

Figures

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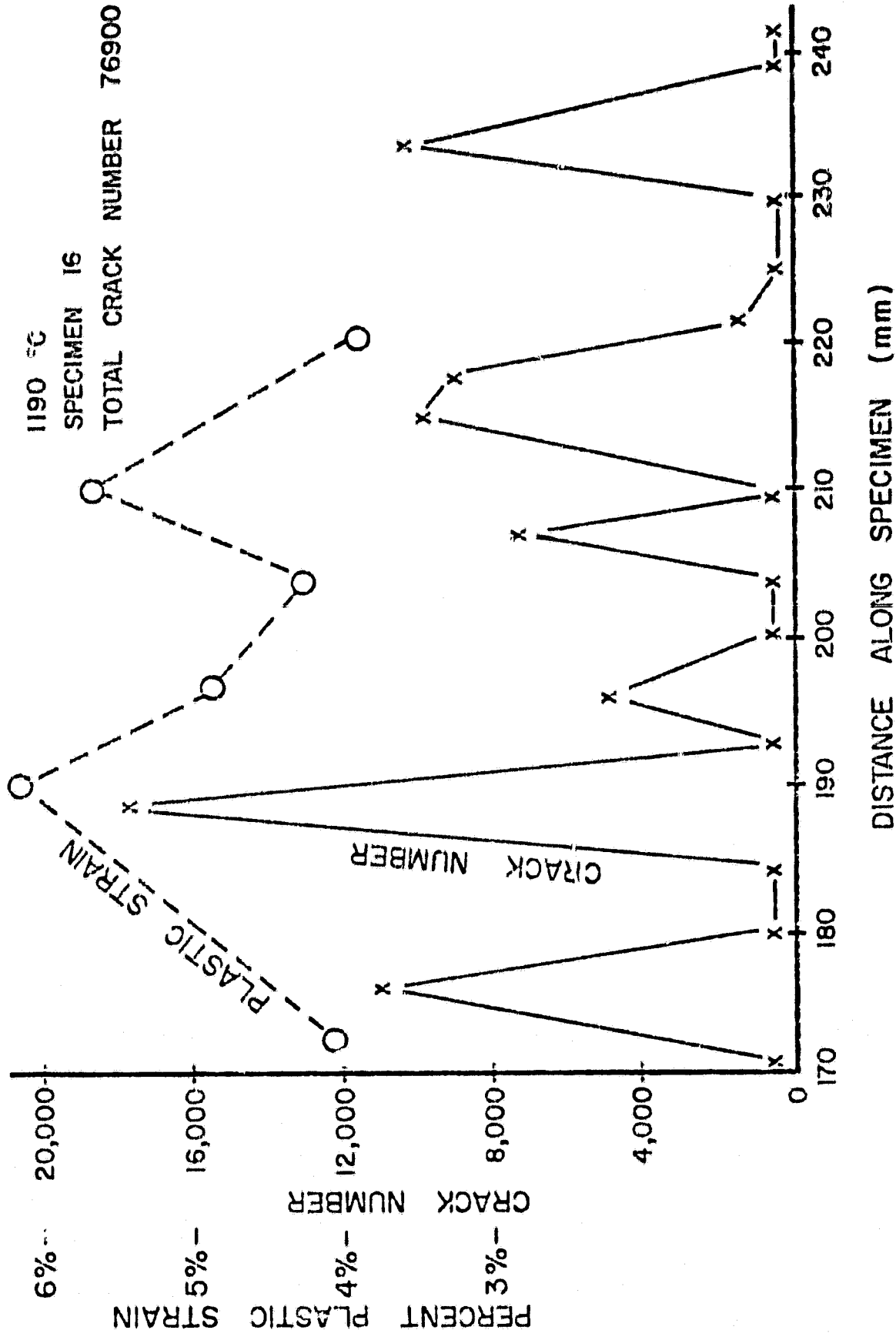


Figure 1. A plot of discrete strain readings along the specimen as compared to crack numbers (N_c) for each four millimeter length of specimen. Evidence indicates scatter in strain due to crack opening displacements.

1150 C

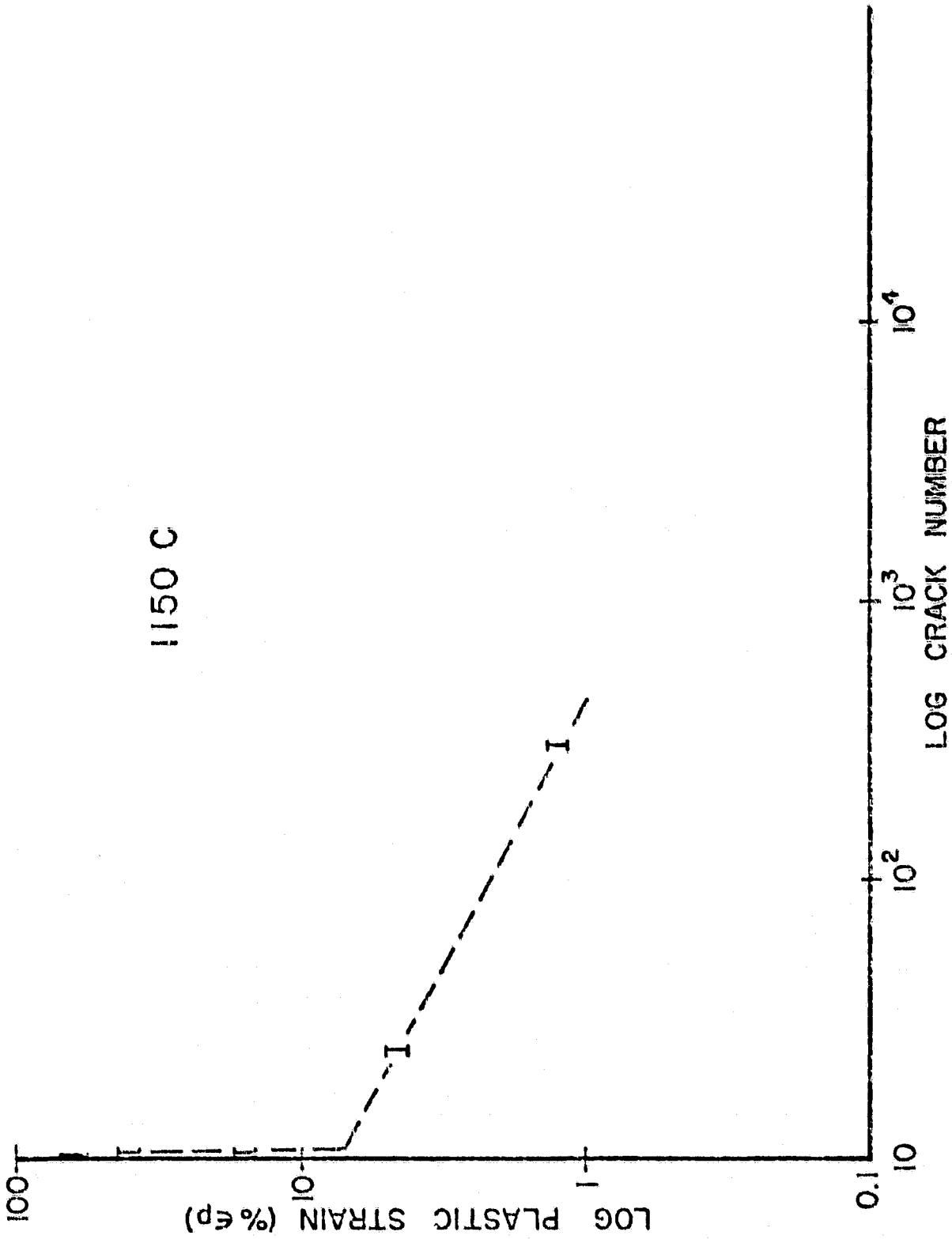


Figure 2. Extrapolation of crack number to the incipient crack ($N_c = 10$).
(a) Extrapolation to incipient crack strain at 1150C.

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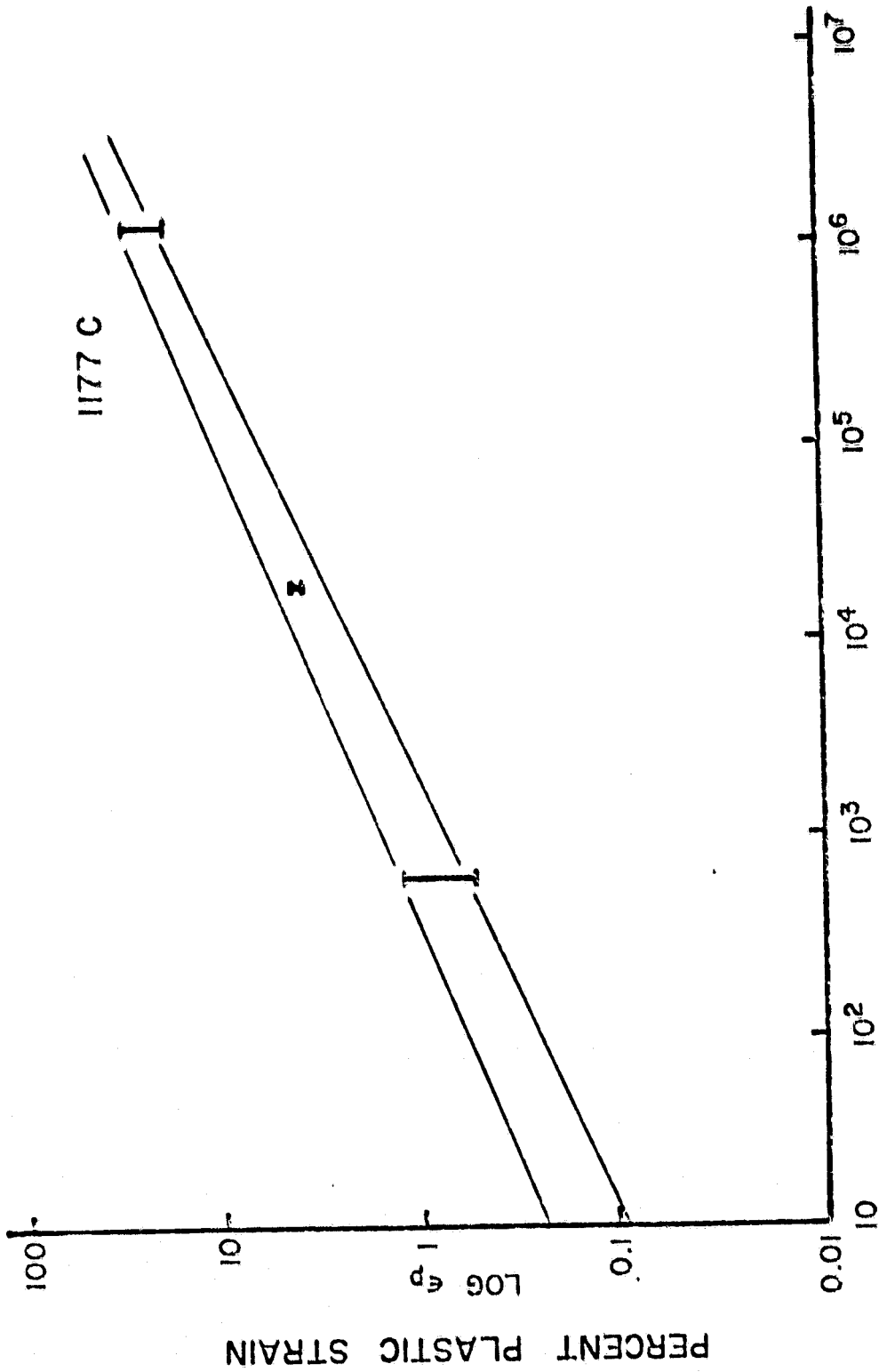


Figure 2. Extrapolation of crack number to the incipient crack ($N_c = 10$).

(b) Extrapolation to incipient crack strain at 1177C.

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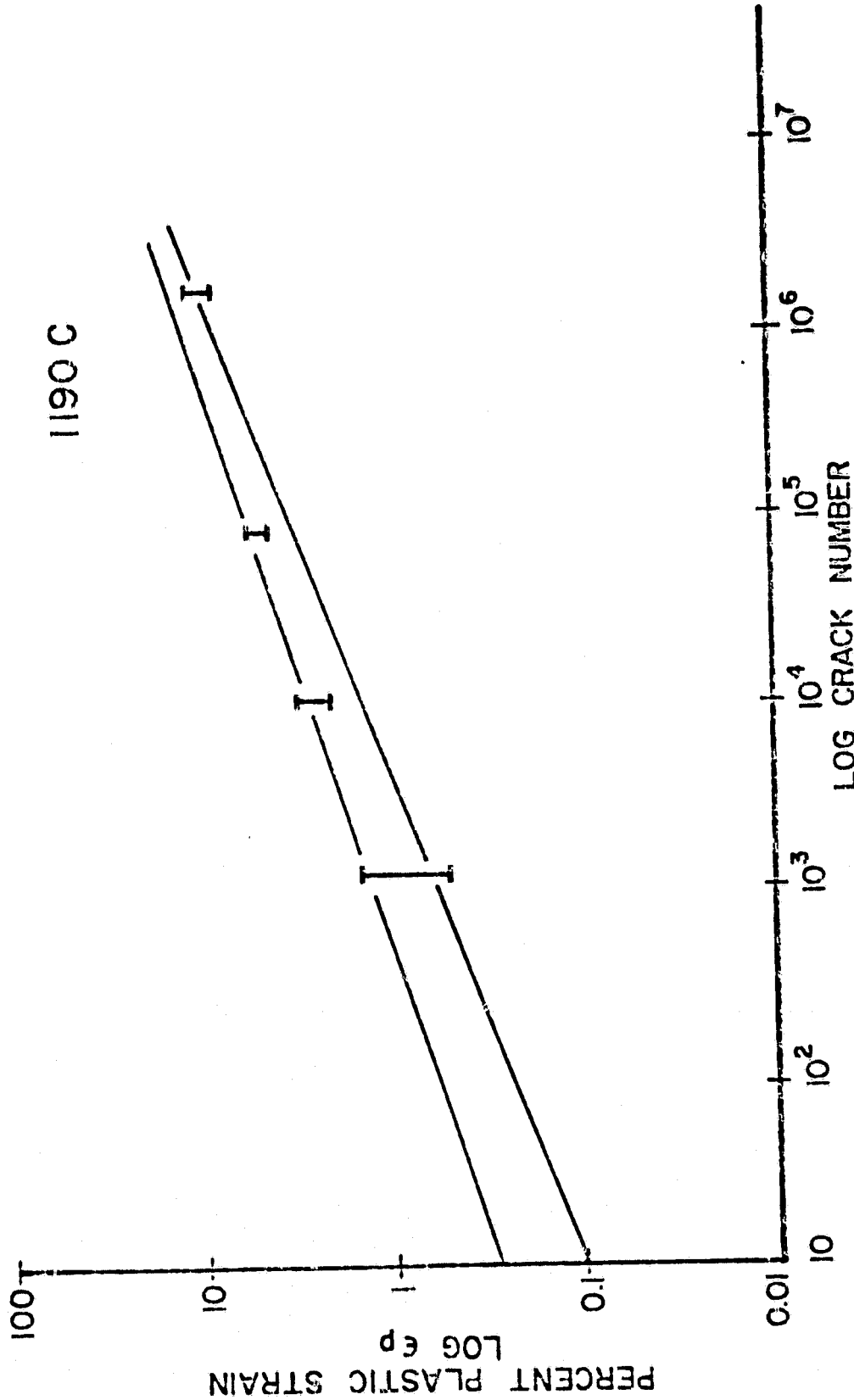


Figure 2. Extrapolation of crack number to the incipient crack ($M_c = 10$).

(c) Extrapolation to incipient crack strain at 1190C.

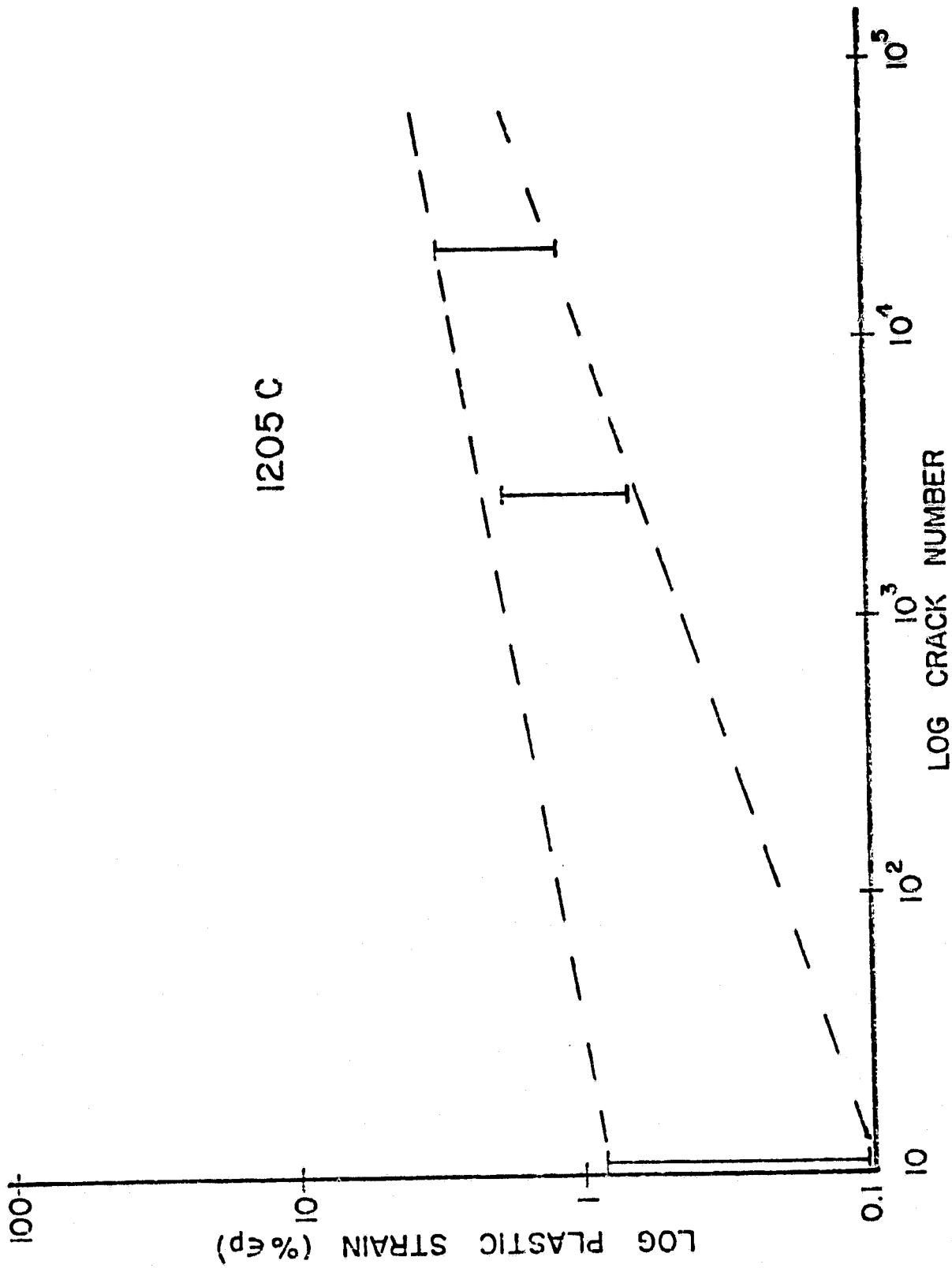
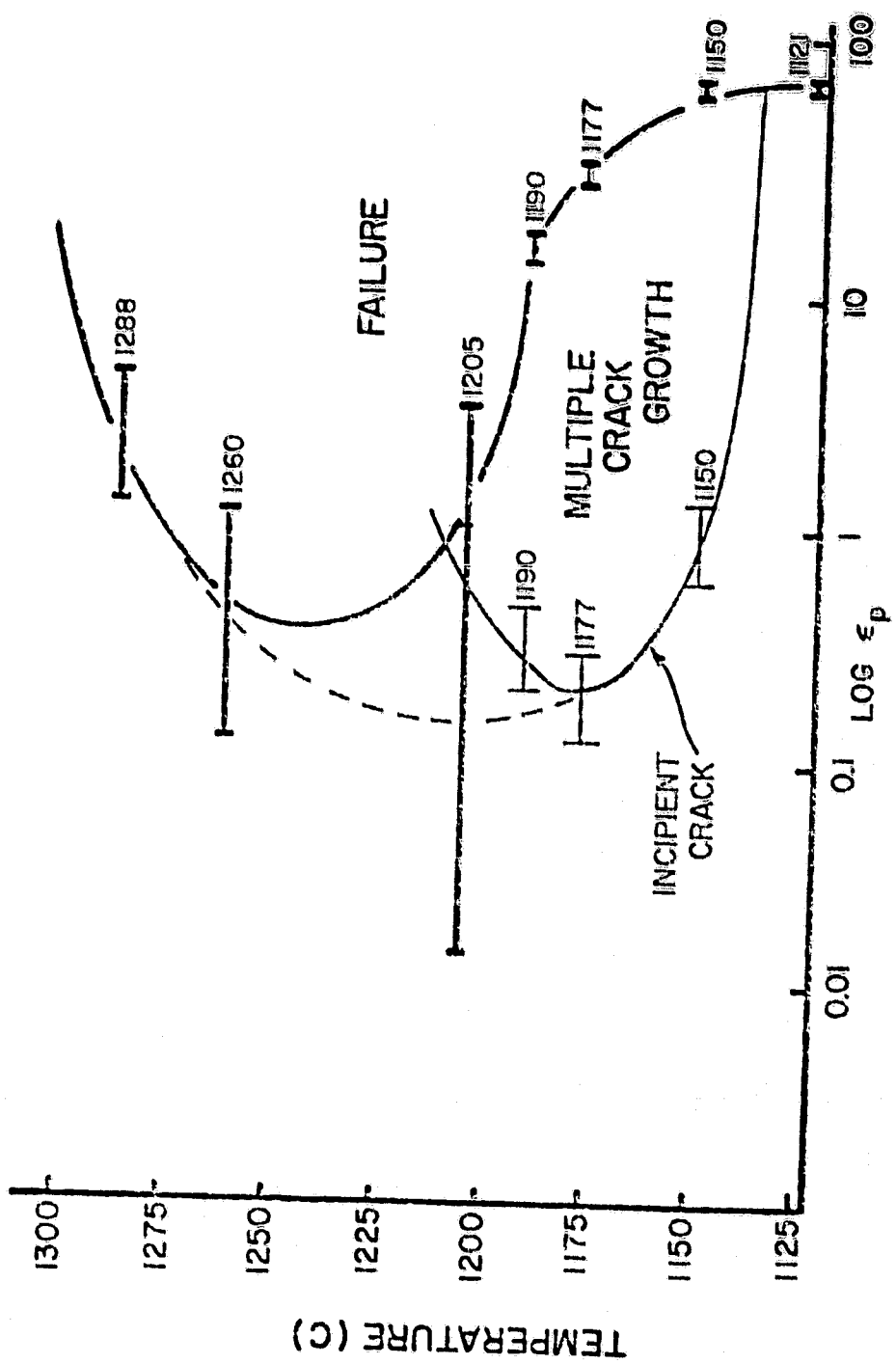


Figure 2. Extrapolation of crack number to the incipient crack ($M_c = 10$).

(d) Extrapolation to incipient crack strain at 1205C.

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PERCENT PLASTIC STRAIN

Figure 3. Incipient cracking envelop as functions of temperature and strain.

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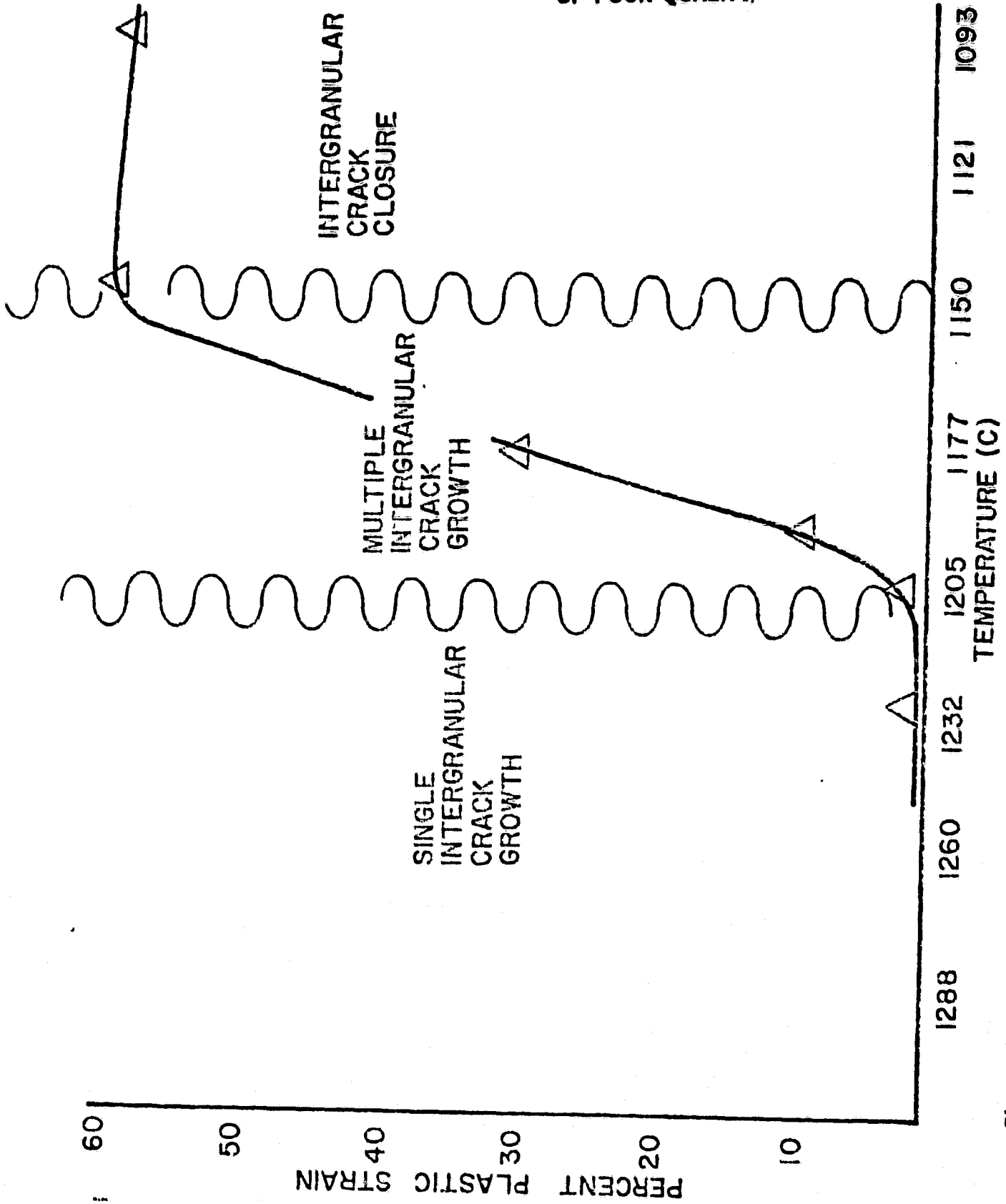


Figure 4. Partition of near-solidus cracking regions in terms of temperature.

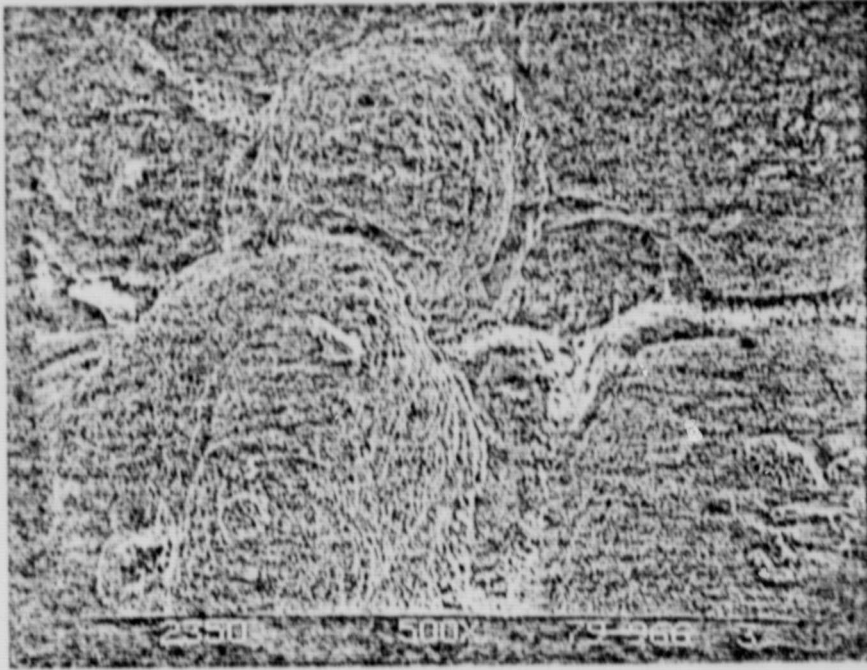


Figure 5. Fracture surface of specimen factured at 1205C.

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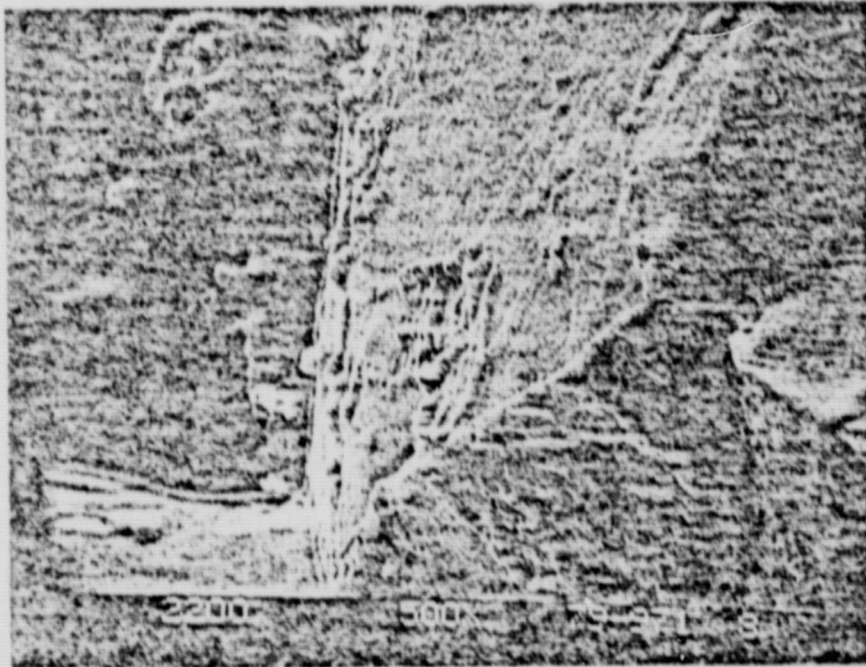


Figure 6. Fracture surface of specimen factured at 1190C.

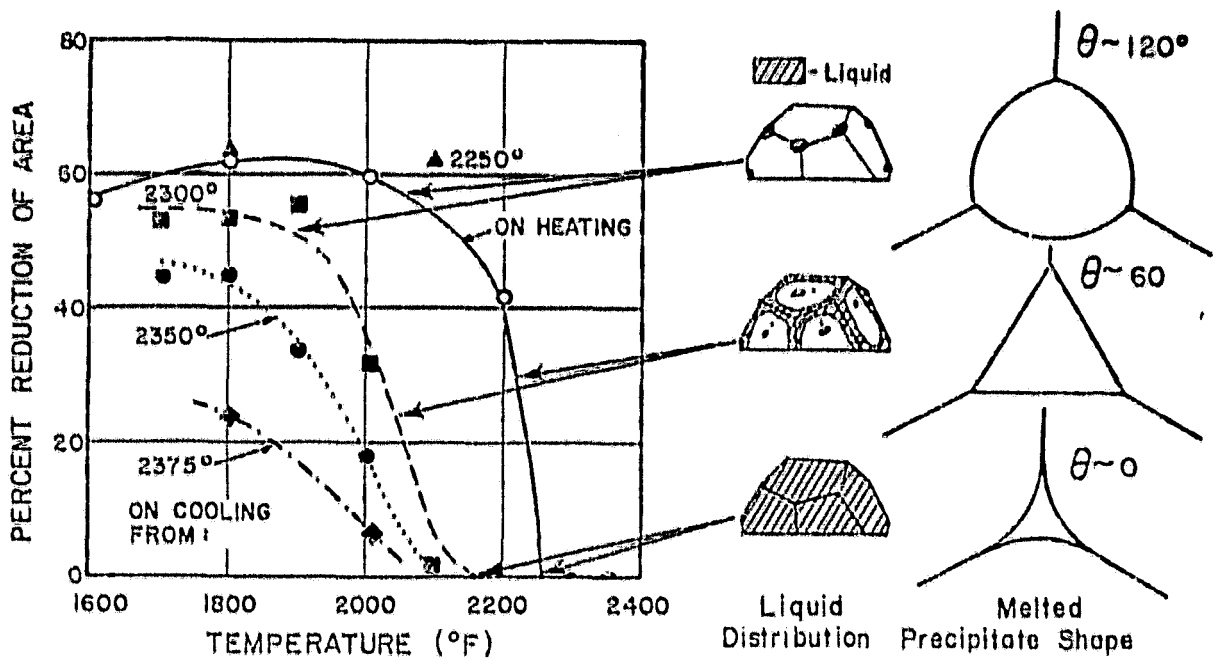


Figure 7. A schematic of partial grain boundary wetting as a function of temperature.

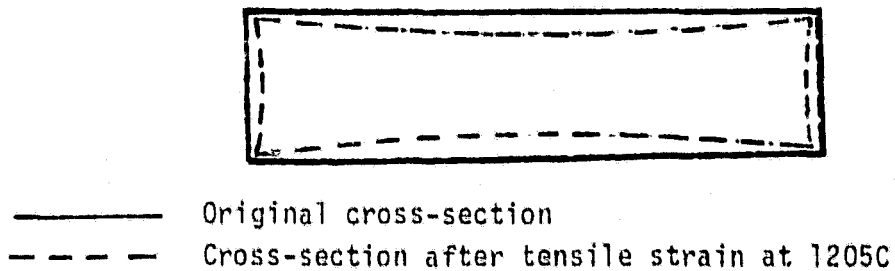


Figure 8. A schematic of change in cross-section due to fluid flow during tensile testing at 1205C.