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Attention: Code SC
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Washington, D. C. 20546

SUBJECT: Progress Report -
"Investigation of the Solidification, Structure and Properties of Eutectic Alloys Including Consideration of Properties Control"

NASA Research Grant NGL-39-007-007

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Submitted by:

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SOLIDIFICATION, STRUCTURE AND PROPERTIES OF EUTECTIC ALLOYS

INCLUDING CONSIDERATION OF PROPERTIES CONTROL

August 1971

This document reports progress made on NASA Grant NGR 39-007-007 since November 1969. In this report, studies of the elevated temperature mechanical behavior of the Ni-Ni_3Nb eutectic composite will be emphasized. Other investigations supported by the Grant (i.e., the Eckelmeyer study of the Mg_2Ni-Mg alloy, Boulbes' study of Bi-MnBi, and the publication of Hoover's papers on Ni-Ni_3Nb) were discussed in my letter of July 20, 1971. In addition, Professor Notis, Assistant Professor of Metallurgy and Materials Science, and an undergraduate student are undertaking a study of the effect of rod size, spacing and composition on superconducting properties of aligned Sn-In eutectic alloy. Also, he is planning another project concerning the physical measurement of elastic moduli of unidirectional solidified eutectic composites. A graduate student on this project will probably begin this study on the Ni-Ni_3Nb system.

ELEVATED TEMPERATURE MECHANICAL BEHAVIOR OF $Ni-Ni_3Nb$

Introduction

Since 1965, two major investigations sponsored by the National Aeronautics and Space Administration (NGR-39-007-007) have been conducted at Lehigh University on the mechanical behavior of the $Ni-Ni_3Nb$ eutectic composite.[1,2] Quinn, employing horizontally grown $Ni-Ni_3Nb$ ingots, surveyed the mechanical properties of the composite from room temperature to 1000°C with hot hardness, hot tensile and stress rupture tests. Quinn used metallographic analysis to tentatively identify a twin-like mode of deformation in the Ni_3Nb phase which was found to operate from room temperature to 1000°C. Fracture of the Ni_3Nb phase was found to control the failure of the composite at all temperatures studied. Quinn reported ultimate tensile strains on the order of 2% at room temperature. Qualitatively, ultimate strain was found to increase slightly as the temperature was raised to 650°C. Above 650°C, a transition to a more brittle type of tensile fracture was noted.

Hoover, employing an improved vertical solidification technique, reported ultimate strains on the order of 15% at room temperature while observing only a slight decrease in strength over those values reported by Quinn. Hoover conclusively identified the tensile deformation mode of the Ni_3Nb as $\{112\}$ type twinning followed by twin boundary cracking. Room temperature results indicated that the unique ductility of the material was directly related to the improved microstructural alignment achieved by vertical solidification.

It is the purpose of the current investigation to characterize the elevated temperature mechanical response of vertically grown $Ni-Ni_3Nb$ by analyzing hot tensile and stress rupture test results. Metallographic analysis will be conducted in hopes of extending current understanding of the composite's deformation and fracture modes to elevated temperatures.

Procedure

Efforts were directed towards unidirectionally solidifying approximately 40 ingots, 9/16 inches diameter by 5 3/4 inches long for the mechanical test program. Master heats were induction melted in an aluminum oxide crucible under a positive pressure of argon. For this study the eutectic composition was taken to be 22.3 w/o Nb. The charge was poured into a steel split mold coated with an MgO mold wash. Difficulties encountered in melting master heats of the material resulted in a two month delay in the progress of the project. Extensive experimentation with casting parameters revealed that the molten charge reacted with chemically combined water in the MgO mold wash. By dehydrating MgO powder at 2000°F for 24 hours and then mixing it in ethyl alcohol instead of water, the problem was eliminated. As-cast pins were unidirectionally solidified in aluminum oxide crucibles under a positive argon pressure at a growth rate of 4.7 cm/hr. Metallographic sections were obtained from each ingot to ensure that controlled microstructures were of eutectic composition and aligned parallel to the ingot axis. According to projected test specimen requirements, the solidification phase of the program has been successfully completed.

Tensile tests have been conducted from room temperature to 1000°C utilizing standard 0.252 inch diameter specimens machined from controlled ingots. Strain was measured at room temperature with an extensometer; however, lack of a high temperature strain gauge limited elevated temperature elongation measurements to load versus total crosshead separation.

Creep rupture tests now in progress are being conducted at constant load, at 600°C, 750°C and 950°C on Reihle and Dennison creep stands outfitted with funds provided during the initial phase of this contract. Creep strains are being measured as a function of time at 600°C with a platinum strip gauge fixed to the one inch gauge length and monitored by a traveling telescope.

Representative fracture surfaces from the hot tensile series have been plated in an electroless nickel bath to guarantee retention of the fracture profile during metallographic preparation. The standard metallographic procedure employed consists of rough polishing through 6 μ and 1 μ diamond paste, and final polishing with a Linde B slurry. Specimens are immersion etched in modified Marbles reagent.

Presentation and Discussion of Preliminary Results

Hot Tensile Results

Figure 1 is a plot of ultimate tensile strength versus testing temperature for aligned Ni-Ni_3Nb tested parallel to the growth direction. Note the initial strength increase between room temperature and 500°C. In this region, ultimate tensile strengths are as much as 25% greater than values reported by Quinn. Also shown are the temperature ranges where deformation response was seen to change. For example, uniform deformation was noted below 600°C while necking occurred above that point. Typical schematic stress strain curves are shown for the uniformly deformed and the necked specimens. Also shown in Figure 1 is the temperature range where serrations were observed in the stress strain curves. Explanations are being sought for those temperature dependent changes in mechanical response.

Figure 2 is a plot of ultimate strain at fracture versus testing temperature. Note the large strains (approximately 40%) reported in the 500°C to 600°C region in sharp contrast to the small values obtained by Quinn (1-5%). The sharp decrease in ultimate strain observed between 700°C and 800°C, is consistent with a corresponding decrease in ultimate strain above 650°C as reported by Quinn.

Metallography

A preliminary metallographic analysis has been conducted on representative hot tensile specimens in an attempt to explain the observed trends in the data. Figure 3a is a typical micrograph of a room temperature fracture profile indicating extensive twin boundary cracking of the Ni_3Nb platelets as reported by Hoover. Figure 3b is a micrograph obtained with polarized light showing $\{112\}$ twins in the intermetallic phase of a room temperature fracture. The presence of twinning, depicted in Figure 3b, is found to be uniformly distributed throughout the gauge length.

Micrographs obtained from the 600°C specimen reveal an apparent transition in deformation mode. Figure 4a shows a longitudinal section of a 600°C fracture surface. Note the absence of cracks in the Ni_3Nb phase and the onset of interfacial delamination at this temperature. Figure 4b is a polarized light micrograph depicting the appearance of twins in the 600°C specimen. The twins appear more diffuse as compared to those observed at room temperature. In addition, the apparent density of twins visible with the light microscope is greatly decreased. The behavior depicted for the 600°C specimen is also observed in 450°C and 560°C specimens. It is hoped that careful metallography, perhaps employing techniques such as phase contrast or electron microscopy, will yield a better picture of the apparent transition in deformation mode between room temperature and 600°C.

Metallographic observation of the 750°C specimen revealed further change in the materials deformation response. Figure 5 is a composite micrograph of a region adjacent to the fracture surface showing deformation markings in the nickel phase and twins in the intermetallic phase. It has been observed that these markings are localized adjacent to the fracture surface, and it appears that they exhibit a crystallographic character. Figure 5 reveals that the markings in the nickel phase are adjacent and parallel to twins in the intermetallic phase. Note that the twins in the Ni_3Nb have reassumed the appearance of room temperature twins. In contrast, however, the lamellar structure depicted in Figure 5 was extensively jogged during deformation, an effect first observed at 750°C (compare Figures 3 and 5). Crystallographic analysis employing known X-ray orientation data and two surface metallography is now in progress to identify the habit planes (if any) of the nickel deformation markings.

Creep Rupture Results

Preliminary results of the creep rupture program are shown in Figure 6. Quinn's data are plotted for comparative purposes. The ultimate strain values obtained in each rupture test are noted in Figure 6. It appears that a ductility transition is occurring with increasing time at 600°C. It is felt that this transition could be related to the ductility transition noted in the hot tensile strain results shown in Figure 2. Elongation measurements obtained during the creep tests have not been analyzed to date.

Future Research

At this point in the program, several avenues for future research are clearly defined. Attention will be directed toward completion of the metallographic examination of the hot tensile specimens in hopes of arriving at an understanding of the complex deformation behavior observed to date. Particular attention will be directed towards understanding the increase in strength and ductility between room temperature and 600°C, the nature of the deformation markings at 750°C, and the causes of both the drop in ductility above 700°C and the secondary increase in ductility above 900°C.

Creep rupture tests currently in progress should be completed by October, 1971. Preliminary data indicates that elongation measurements at 600°C will provide valuable phenomenological data pertaining to the effects of stress and rupture life on the minimum creep rate of the composite.

It is projected that metallographic analysis of creep rupture specimens, when combined with results of the hot tensile study, will yield valuable insights into understanding the effects of time and temperature on the ductility transition which appears to be occurring during both tensile and creep rupture testing.

References

1. R. T. Quinn, Structure and Elevated Temperature Mechanical Behavior of Unidirectionally Solidified Ni-Ni_3Nb Eutectic Alloy, Ph.D. Dissertation, Lehigh University, (1967).

2. W. R. Hoover, The Monotonic and Cyclic Mechanical Response of the Ni-Ni_3Nb Eutectic Composite, Ph.D. Dissertation, Lehigh University, (1970).

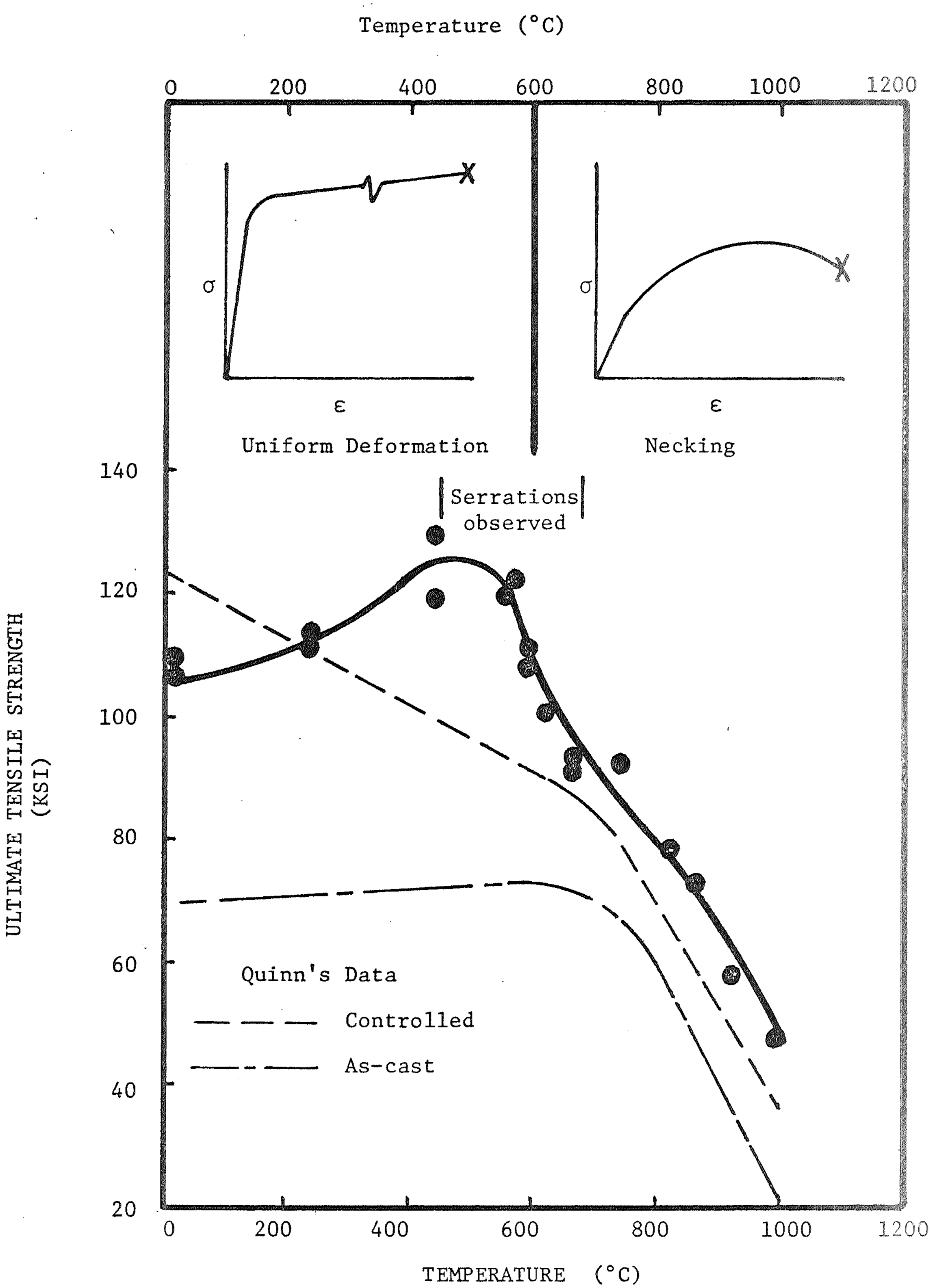


Figure 1: The Ultimate Tensile Strength of Unidirectionally Solidified $Ni\text{-}Ni_3Nb$ Measured as a Function of Temperature

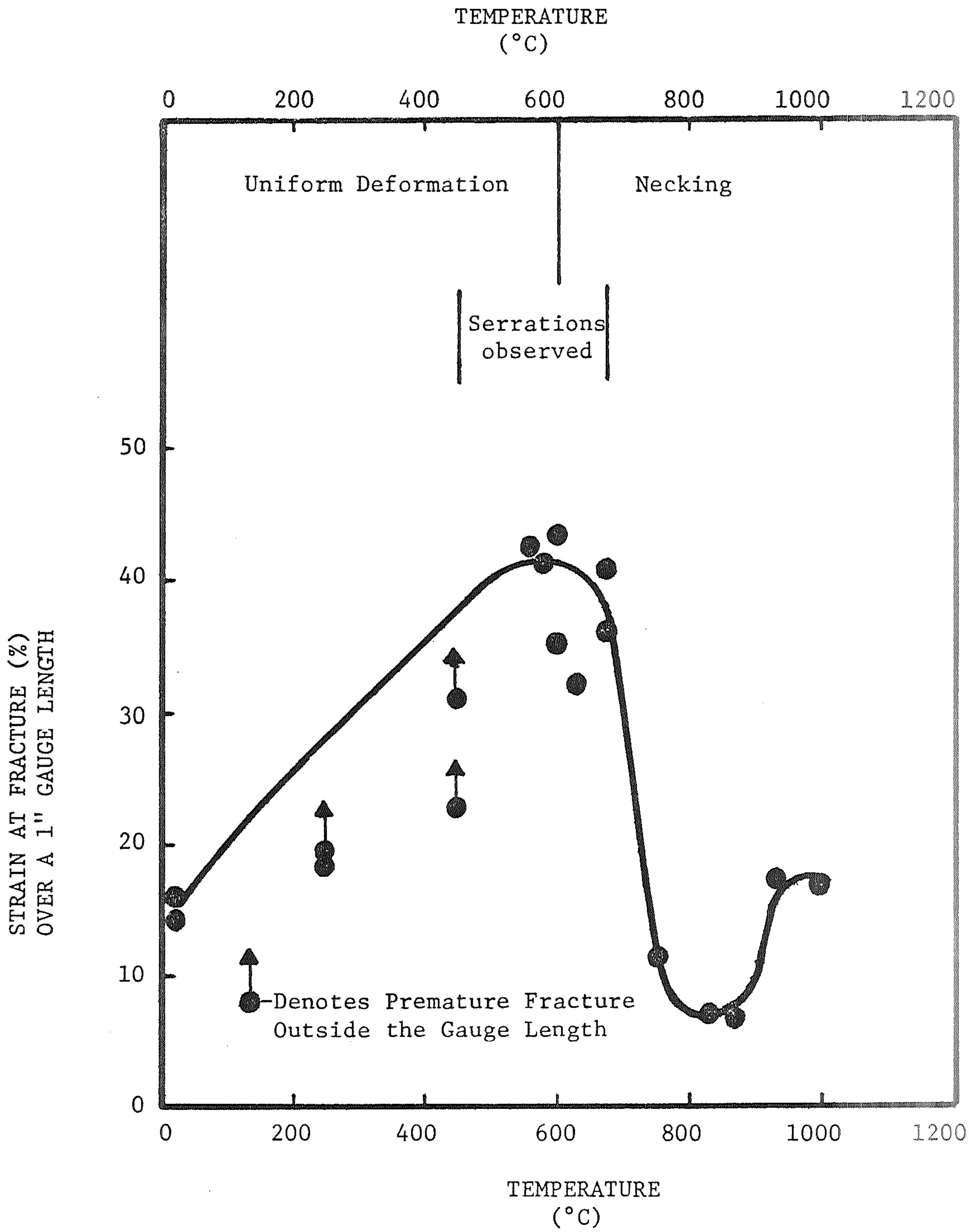
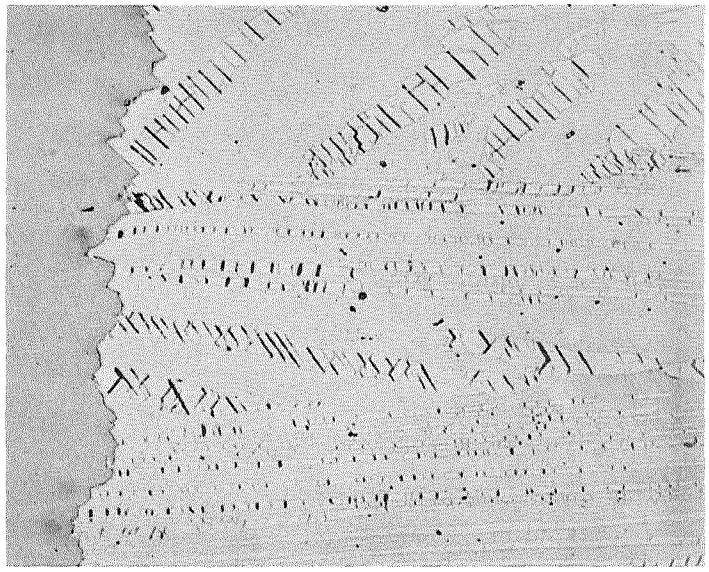


Figure 2: Ultimate Strain At Fracture As A Function Of Test Temperature.



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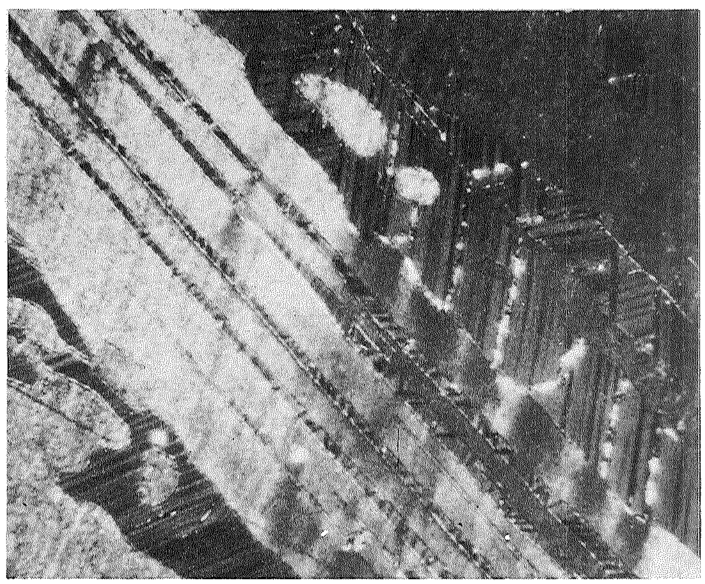

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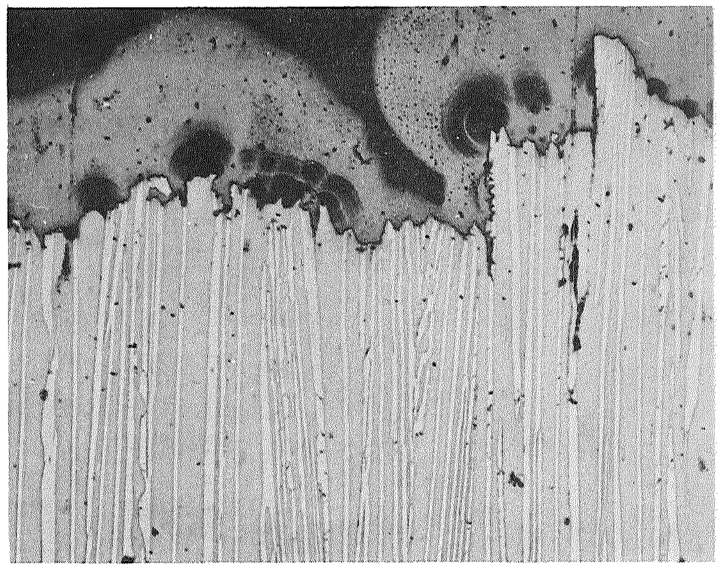
Figure 3. a) Longitudinal profile of a room temperature tensile fracture. (The Ni phase is grey while the Ni_3Nb phase is white), 200X.
b) Polarized light micrograph of {112} type twins in Ni_3Nb deformed in tension at room temperature, 530X.



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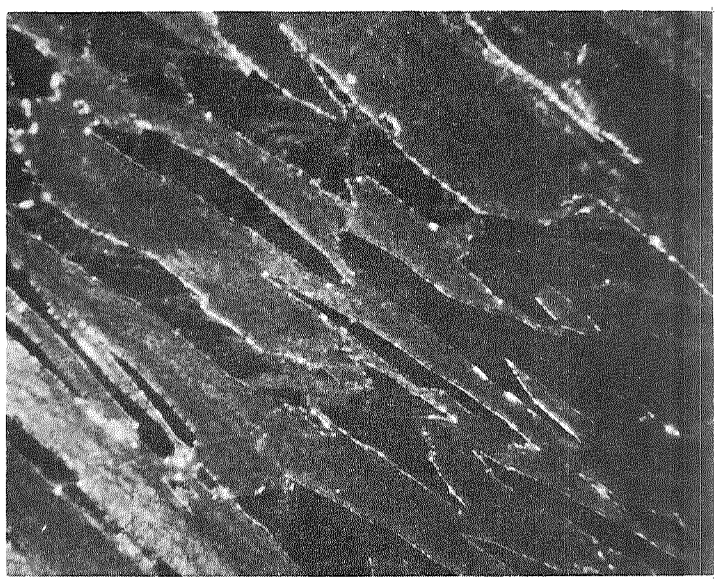

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Figure 4. a) Longitudinal profile of a 600° C tensile fracture, 200X.
b) Polarized light micrograph obtained adjacent to a 600° C tensile fracture depicting twins in the Ni_3Nb phase, 530X.

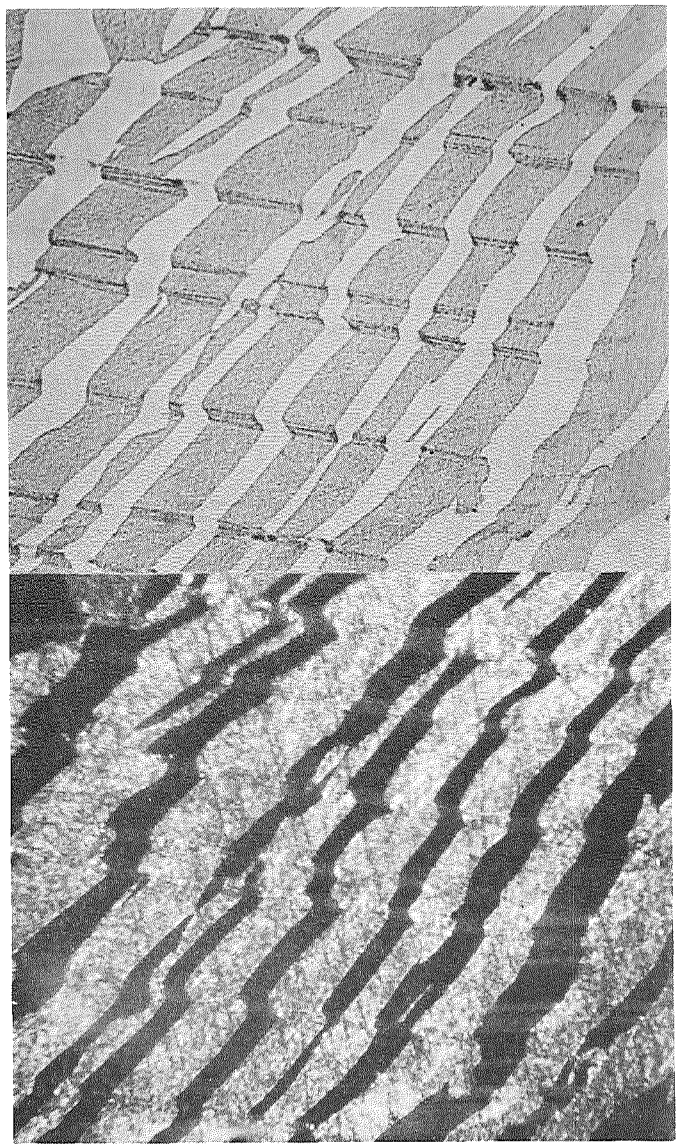

Figure 5. White and polarized light micrographs of the same region adjacent to a 750° C tensile fracture revealing the relation between Ni_3Nb twins and Ni deformation markings. (Ni is the dark phase as viewed in white light), 530X.

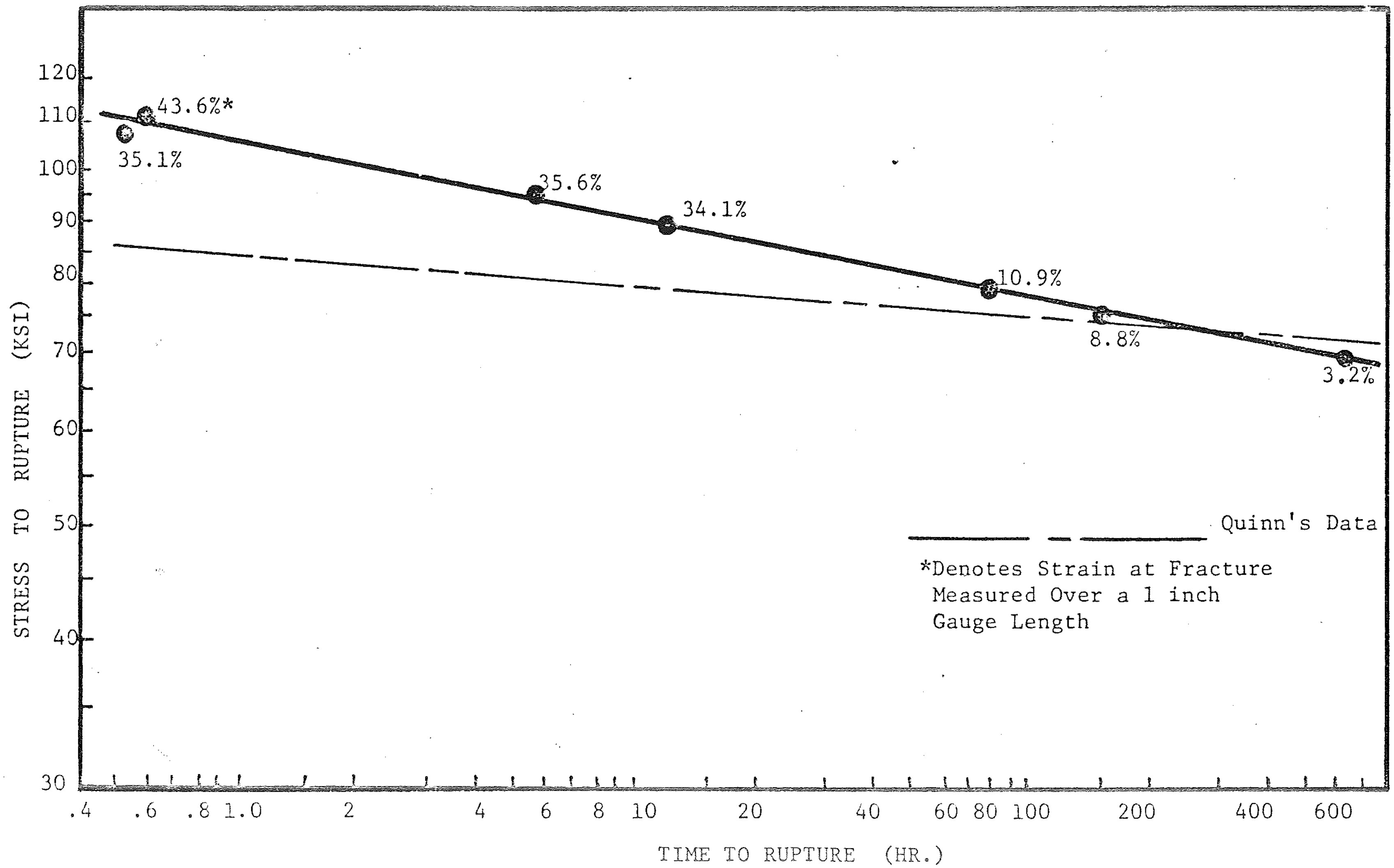

Figure 6: 600°C Stress Rupture Data for Unidirectionally Solidified $Ni-Ni_3Nb$