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G. E. Fuchs

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KAPL ATOMIC POWER LABORATORY

SCHENECTADY, NEW YORK 10701

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## THE HOT WORKABILITY AND SUPERPLASTICITY OF Ti-48Al-2Nb-2Cr ALLOYS

G.E. Fuchs  
Lockheed Martin Corporation  
P.O. Box 1072  
Schenectady, NY 12301-1072

### Abstract

The hot compression behavior and microstructure evolution of ingot metallurgy (I/M) and powder metallurgy (P/M) processed samples of the near- $\gamma$  Ti-aluminide alloy, Ti-48Al-2Nb-2Cr (at%), were determined. Three I/M conditions and two P/M conditions were examined in this study. Hot compression tests were performed in the temperature range 1100°-1300°C at strain rates ranging from  $1.67 \times 10^{-1}$ /sec to  $1.67 \times 10^{-4}$ /sec. P/M materials consolidated by either hot isostatic pressing (HIP'ing) or extrusion exhibited the best hot workability in most cases. The P/M materials possessed finer, more homogeneous microstructures than the I/M materials. It was also noted that improved workability, and in some cases superplastic behavior, was observed in the materials with equiaxed microstructures without any lamellar constituents.

## Introduction

Two-phase, near- $\gamma$  titanium aluminides, such as Ti-48Al-2Nb-2Cr (at%), are being considered for high temperature structural applications in the aerospace and automotive industries due to their high specific strength and good oxidation resistance at elevated temperatures (1-4). Near- $\gamma$  alloys with increased toughness and ductility have been developed by understanding the effects of alloying additions and processing on the microstructure and properties (4,5).

Although hot working by isothermal forging and extrusion have been widely used by many investigators to study microstructural development, there are only a limited number of reports on the hot working behavior of  $\gamma$  titanium aluminides (6-20). These reports on hot workability have focussed on ingot metallurgy (I/M) processing and typically emphasized one material starting condition (14, 21-27). This study examined the effect of processing on the hot workability of a single alloy composition.

## Experimental Procedures

Materials with a nominal composition of Ti-48Al-2Nb-2Cr (unless otherwise noted, all compositions are in atomic %) were prepared by both ingot metallurgy and powder metallurgy techniques (Tables 1 and 2).

The I/M samples were prepared from a single 20 Kg vacuum induction skull melted ingot prepared by Duriron Co., Inc. (Dayton, OH). The ingot (ID TA-22) measured 7 cm diameter x 75 cm. The ingot was HIP'ed at 1260°C/172MPa/4hrs to eliminate any residual casting shrink or porosity. The ingot was then sectioned into 25 cm lengths and heat treated to homogenize the as-cast+HIP microstructure. The homogenization heat treatments utilized in this study were based on previous studies (24). A near- $\gamma$  microstructure consisting of equiaxed  $\gamma$  grains with small amounts of equiaxed  $\alpha_2$  and  $\beta$  grains was produced by heat treatment at 1200°C for 96 hrs (ID TA-22A). A significant amount of coarse twins were observed in the  $\gamma$  grains. A limited amount of  $\gamma/\alpha_2$  lamellar grains were observed in the TA-22A samples which were the remnants of the prior  $\alpha$ -dendrites in the as-cast microstructure. Heat treatment of the samples at 1440°C for 20 minutes resulted in a coarse grained lamellar microstructure (ID TA-22C).

Some of the samples given the 1440°C/20min homogenization heat treatment were isothermally forged at 1175°C/1.67x10<sup>-3</sup>sec<sup>-1</sup>/30% (ID TA-22X). The as-forged samples exhibited a limited amount of dynamic recrystallization. Complete recrystallization was observed when the forged samples were heat treated at 1200°C/48hrs. The near- $\gamma$  recrystallized microstructure consisted of relatively fine equiaxed  $\gamma$  grains with a limited amount of equiaxed  $\alpha_2$  and  $\beta$ .

The powder metallurgy materials were prepared from a single 20 Kg argon gas atomized heat produced at Crucible Research Center (Pittsburgh, PA). The powder was sieved to a -35 mesh size (<500 $\mu$ m). Approximately half of the powder yield was canned in a commercial purity Ti (CP-Ti) can with approximate dimensions of 10 cm dia x 30 cm, evacuated and HIP'ed at 1230°C/172MPa/4hrs (ID TA-47). The as-HIP material exhibited a near- $\gamma$  microstructure predominantly comprised of equiaxed  $\gamma$  grains with annealing twins and a limited amount of equiaxed  $\alpha_2$  and  $\beta$  grains. The remaining powder was consolidated by extrusion (1300°C/16:1) and slow cooled in vermiculite

sand (TA-50). A fine grained duplex microstructure with equiaxed  $\gamma$ ,  $\alpha_2$  and  $\beta$  grains and limited amounts of lamellar  $\gamma/\alpha_2$  grains was observed in the extruded sample.

Hot compression samples were prepared from the five starting conditions (3 I/M and 2 P/M) listed in Table 2. The right cylindrical samples were ground and had dimensions of 12.7 cm dia x 19 cm. Grooves were machined into the parallel ends of each sample to retain lubricant during the compression testing. Constant strain rate, isothermal hot compression testing was performed at the University of Pittsburgh (Pittsburgh, PA) in a servo-hydraulic mechanical testing machine. The samples were tested on SiC platens in a flowing argon environment. The sample/platen interface was lubricated with boron nitride. The test temperatures were 1100°, 1150°, 1175°, 1250° and 1300°C. The sample heating and thermal stabilization prior to upsetting took approximately 30 minutes. The strain rates evaluated included  $1.67 \times 10^{-1}/\text{sec}$ ,  $1.67 \times 10^{-2}/\text{sec}$ ,  $1.67 \times 10^{-3}/\text{sec}$  and  $1.67 \times 10^{-4}/\text{sec}$ . Samples were compressed to a final height of 9.5 cm for a height reduction of 70% true strain (50% height reduction). Following compression testing, the samples were rapidly cooled to room temperature by forced convection cooling.

After compression testing, the samples were sectioned by water-jet cutting and prepared for microstructural evaluation. Metallographic samples were mechanically polished and etched in Kroll's reagent and examined by optical metallographic and scanning electron microscopy (SEM) techniques.

### Results and Discussion

The measured flow stress for all of the samples exhibited a very strong dependence on the test temperature and strain rate (Figure 1). Processing also had a significant effect on the flow stress (Figure 1). Several different types of flow curves were observed (Table 3). In most cases, flow softening was observed after a sharp yield peak or after a general yielding. At low strain rates and high test temperatures, all of the flow softening was attributed to microstructural changes during the deformation. At high strain rates and low test temperatures, a significant amount of flow softening was ascribed to deformation heating and the large temperature dependence of the flow stress. To differentiate between flow softening resulting from deformation heating and that arising from microstructural changes, techniques to estimate adiabatic heating to correct flow stress curves were utilized (10, 17, 19).

A constant flow stress behavior was observed in some cases at intermediate to low strain rates. These constant flow curves are generally ascribed to a balance between dynamic recrystallization, other microstructural changes and work hardening due to deformation. Lastly, at high test temperatures and slow strain rates, a limited amount of flow hardening was observed in two samples (P/M HIP and extruded) due to grain growth.

### I/M Materials

The peak flow stress ( $\sigma_{\text{PFS}}$ ) for each of the three I/M sample conditions is presented in Figure 2. Note that for the test conditions evaluated in this work, the TA-22C samples, which possessed the coarse grained lamellar microstructures exhibited the highest peak flow stresses ( $\sigma_{\text{PFS}}$ ), but the lowest steady state flow stresses of the I/M samples. The samples that were forged and recrystallized (TA-22X) and the samples that were homogenized at low temperatures (TA-22A) exhibited the lowest peak flow stresses ( $\sigma_{\text{PFS}}$ ) and relatively low steady state flow stresses. None of the I/M samples exhibited

any evidence of external cracking.

Flow softening due to dynamic recrystallization was generally observed in the TA-22A and TA-22X samples at temperatures up to 1175°C at high strain rates (Figure 3a). Decreasing the strain rates and increasing the test temperatures resulted in a balance between dynamic recrystallization and work hardening. However, shear instabilities were observed in the TA-22C and TA-22X samples tested at the highest test temperature and the lowest strain rate. At the higher test temperatures (i.e., 1250°C and 1300°C), flow softening was observed at the highest strain rates. In contrast, flow softening due to dynamic recrystallization was observed in virtually all of the test conditions evaluated in the TA-22C samples (Figure 3c).

The strain rate sensitivity ( $m$ ) was observed to increase with increasing test temperature to a peak at approximately 1250°C (Figure 4). A slight decrease in the strain rate sensitivity was observed at 1300°C. The I/M materials did appear to approach superplasticity based on the strain rate sensitivity ( $m > 0.3$ ). Among the I/M materials, the TA-22X samples, which were forged and recrystallized, exhibited the highest levels of strain rate sensitivity,  $m$ , and at 1250°C exhibited a strain rate sensitivity greater than 0.30. The TA-22C samples with the coarse grained lamellar microstructure, exhibited the lowest level of strain rate sensitivity and the TA-22A samples had intermediate levels of strain rate sensitivity.

The apparent activation energy for the deformation processes of the I/M samples were determined at a fixed stress to be in the range of 308 to 356 KJ/mol. These activation energies are close to values reported for bulk diffusion in Ti-48Al (6,7,10, 17).

### P/M Materials

The peak flow stress ( $\sigma_{PFS}$ ) for both of the P/M materials were generally lower than that of the I/M samples (Figure 2). The HIP'ed samples exhibited slightly greater flow stresses than the fine grained extruded material. Severe cracking occurred during high strain rate compression testing of the HIP'ed materials at 1100°C. Increasing the temperature to 1150°C resulted in reduced cracking. Further increasing the test temperature to 1175°C and/or decreasing the strain rate completely eliminated the occurrence of cracking. No evidence of cracking was observed in the extruded sample.

The majority of the flow curves from the HIP'ed and the extruded samples were either flow softening or constant flow stress due to dynamic recrystallization. At 1250°C, flow hardening was observed at the slowest strain rate due to grain growth (Figure 3).

The strain rate sensitivity,  $m$ , determined for the P/M samples was significantly higher than those determined for the I/M samples (Figure 4). The HIP'ed materials (TA-47) exhibited a strain rate sensitivity,  $m$ , greater than 0.3 at 1250°C and 1300°C. In fact, the HIP'ed material exhibited the greatest level of strain rate sensitivity and the greatest potential for superplasticity ( $m = 0.328$ ) of any of the samples tested. The finer grained extruded material (TA-50) exhibited strain rate sensitivity,  $m$ , level similar to that of the HIP'ed material, but only exhibited  $m$ -values greater than 0.3 at 1300°C.

The apparent activation energy was determined for a fixed stress for both P/M materials was determined to be 308-310 KJ/mol. This value of activation energy is, again, similar to values reported for bulk diffusion in Ti-48Al (6, 7, 10, 17).

### I/M versus P/M

In general, the P/M samples exhibited lower flow stresses and greater strain rate sensitivities than the I/M samples. Although this difference in flow behavior, in part, may be due to grain size, additional microstructural features appear to be at least as important as the grain size. The finer grain size did appear to result in lower flow stresses, reduced flow softening and greater strain rate sensitivity. However, the presence of lamellar  $\gamma/\alpha$  or  $\gamma/\alpha_2$  appeared to result in reduced strain rate sensitivity and increased amounts of flow softening and higher flow stresses. The lower flow stresses, increased strain rate sensitivity and reduced flow softening, observed in equiaxed, fine grained microstructures, would be expected to result in improved hot workability, the potential for superplastic forming and perhaps reduced processing cost. The desired microstructure can be produced in Ti-48Al-2Nb-2Cr alloys by HIP consolidation of P/M alloys and by repeated hot working and recrystallization of I/M materials.

### Summary

The elevated temperature flow behavior was determined for 3 I/M and 2 P/M TiAl-based alloys in the temperature range 1100°-1300°C. Increasing the test temperature resulted in increased strain rate sensitivity and reduced flow stresses. Superplasticity was indicated for the forged and recrystallized I/M samples (TA-22X) at 1250°C, the HIP'ed P/M material (TA-47) at 1250°-1300°C and at 1300°C for the extruded P/M material (TA-50). Refined grain size and equiaxed phase morphologies appeared to result in increased strain rate sensitivity, superplastic behavior and reduced processing costs.

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**Table 1**

Composition in Atomic % of Ti-48Al-2Nb-2Cr alloys

<u>Element</u>	<u>TA-22 (I/M)</u>	<u>TA-47/50 (P/M)</u>
Ti	Bal	Bal
Al	47.25	47.73
Nb	1.96	1.77
Cr	2.04	1.99
C	0.047	0.032
O	0.156	0.136
H	0.070	0.061
N	0.019	0.006

**Table 2**

Processing History and Starting Grain Size of Ti-48Al-2Nb-2Cr Alloys

<u>Alloy ID</u>	<u>Processing History</u>	<u>Grain Size (<math>\mu\text{m}</math>)</u>
TA-22A	I/M: Cast + HIP(1260°C/172MPa/4hrs) + 1200°C/96hrs	115
TA-22C	I/M: Cast + HIP(1260°C/172MPa/4hrs) + 1440°C/20min	1050
TA-22X	I/M: Cast + HIP(1260°C/172MPa/4hrs) + 1440°C/20min + Isothermal Forge (1175°C/1.67x10 <sup>-3</sup> sec <sup>-1</sup> /30%) + 1200°C/48hrs	45
TA-47	P/M: HIP(1230°C/172MPa/4hrs)	22
TA-50	P/M: Extrude(1300°C/16:1/Sand Cool)	10

**Table 3**

## Hot Compression Flow Curve Type

(FS = Flow Soften, CF = Constant Flow, FH = Flow Harden)

**1100°C**

<u>Alloy ID</u>	<u>1.67x10<sup>-1</sup>/sec</u>	<u>1.67x10<sup>-2</sup>/sec</u>	<u>1.67x10<sup>-3</sup>/sec</u>	<u>1.67x10<sup>-4</sup>/sec</u>
TA-22A	FS	FS	CF	CF
TA-22C	FS	FS	FS	FS
TA-22X	FS	FS	CF	CF
TA-47	Fractured	FS	FS	CF
TA-50	FS	FS	CF	CF

**1150°C**

<u>Alloy ID</u>	<u>1.67x10<sup>-1</sup>/sec</u>	<u>1.67x10<sup>-2</sup>/sec</u>	<u>1.67x10<sup>-3</sup>/sec</u>	<u>1.67x10<sup>-4</sup>/sec</u>
TA-22A	FS	CF	CF	CF
TA-22C	FS	FS	FS	FS
TA-22X	FS	CF	CF	CF
TA-47	FS	FS	CF	CF
TA-50	FS	FS	CF	CF

**1175°C**

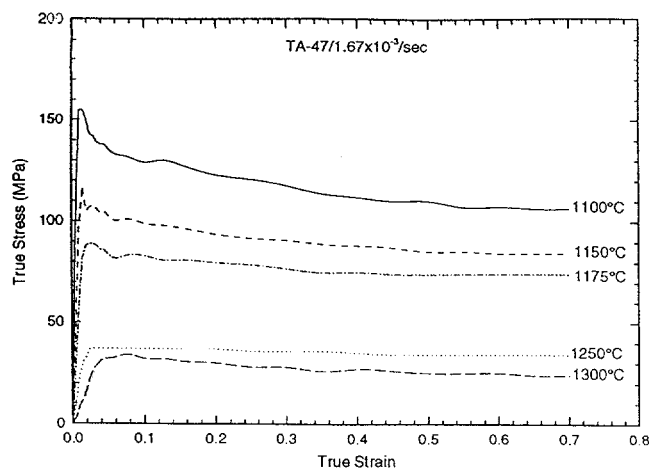
<u>Alloy ID</u>	<u>1.67x10<sup>-1</sup>/sec</u>	<u>1.67x10<sup>-2</sup>/sec</u>	<u>1.67x10<sup>-3</sup>/sec</u>	<u>1.67x10<sup>-4</sup>/sec</u>
TA-22A	FS	CF	CF	CF
TA-22C	FS	FS	FS	FS
TA-22X	FS	CF	CF	CF
TA-47	FS	FS	CF	CF
TA-50	FS	FS	CF	CF

**1250°C**

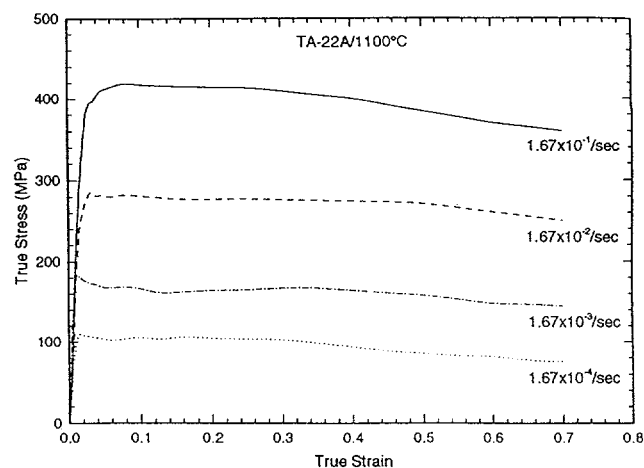
<u>Alloy ID</u>	<u>1.67x10<sup>-1</sup>/sec</u>	<u>1.67x10<sup>-2</sup>/sec</u>	<u>1.67x10<sup>-3</sup>/sec</u>	<u>1.67x10<sup>-4</sup>/sec</u>
TA-22A	FS	CF	CF	CF
TA-22C	FS	FS	FS	CF
TA-22X	FS	CF	CF	CF
TA-47	FS	CF	CF	FH
TA-50	FS	CF	CF	FH

**1300°C**

<u>Alloy ID</u>	<u>1.67x10<sup>-1</sup>/sec</u>	<u>1.67x10<sup>-2</sup>/sec</u>	<u>1.67x10<sup>-3</sup>/sec</u>	<u>1.67x10<sup>-4</sup>/sec</u>
TA-22A	FS	FS	FS	FS
TA-22C	FS	FS	FS	Excessive shear
TA-22X	FS	FS	FS	Excessive shear
TA-47	FS	FS	FS	CF
TA-50	FS	FS	FS	CF



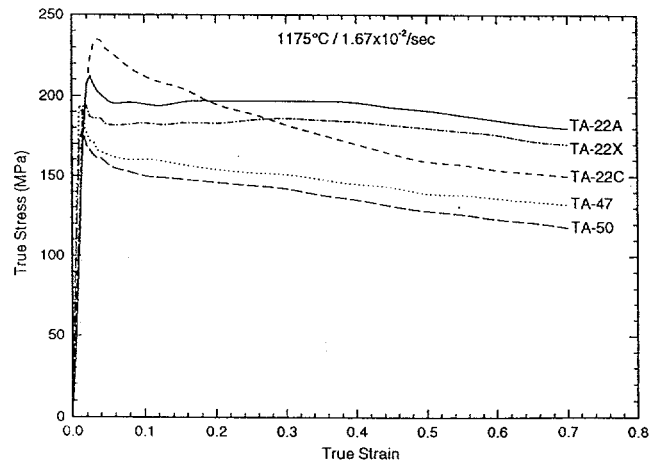
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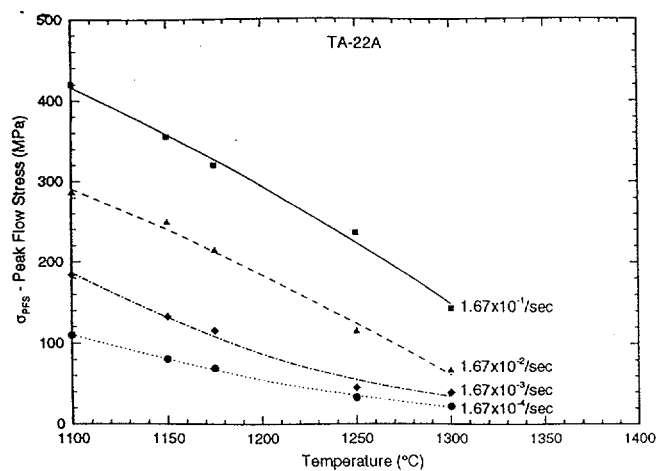
b.)

Figure 1 - Effects on temperature, strain rate and processing on the flow stress behavior of Ti-48Al-2Nb-2Cr alloys. Note that the flow curves have been corrected for adiabatic heating. Alloys TA-47 and TA-50 are P/M process samples and TA-22A, TA-22C and TA-22X are I/M samples.

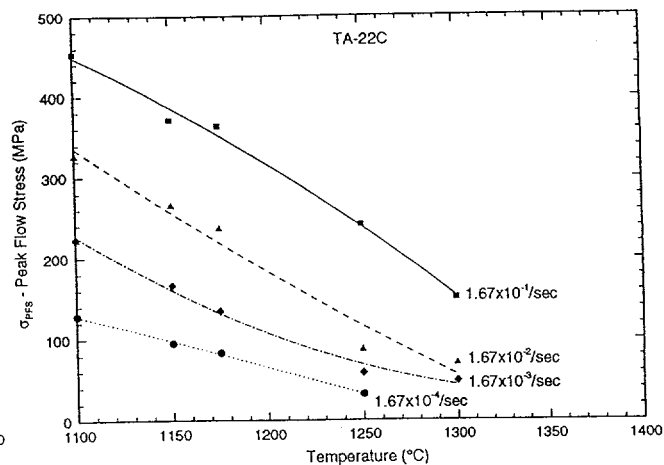
- a.) Effect of Temperature
- b.) Effect of Strain Rate
- c.) Effect of Processing



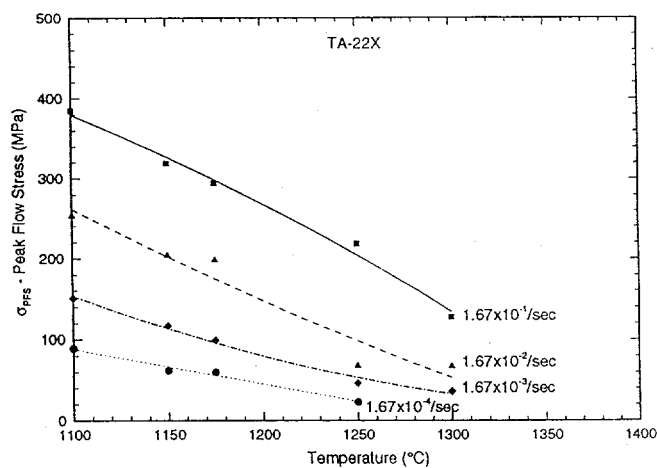
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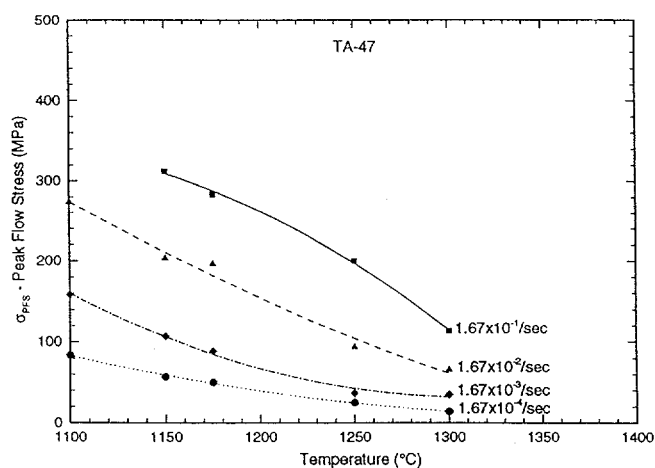
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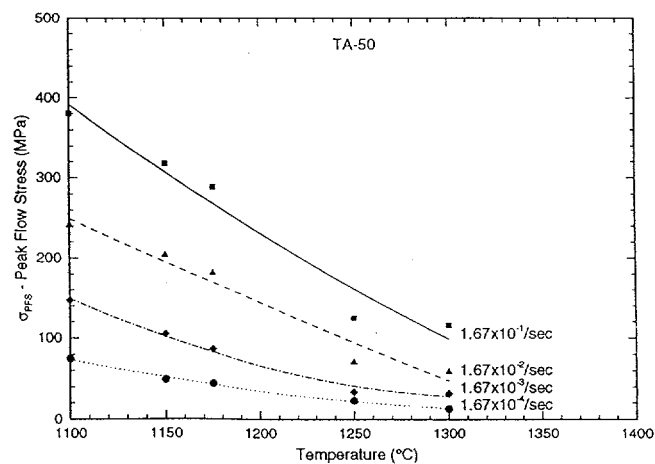
b.)



c.)



d.)



e.)

Figure 2 - The effect of temperature on the peak flow stress ( $\sigma_{PFS}$ ) on the isothermal compression tested I/M and P/M Ti-48Al-2Nb-2Cr samples.

- a.) TA-22A
- b.) TA-22C
- c.) TA-22X
- d.) TA-47
- e.) TA-50

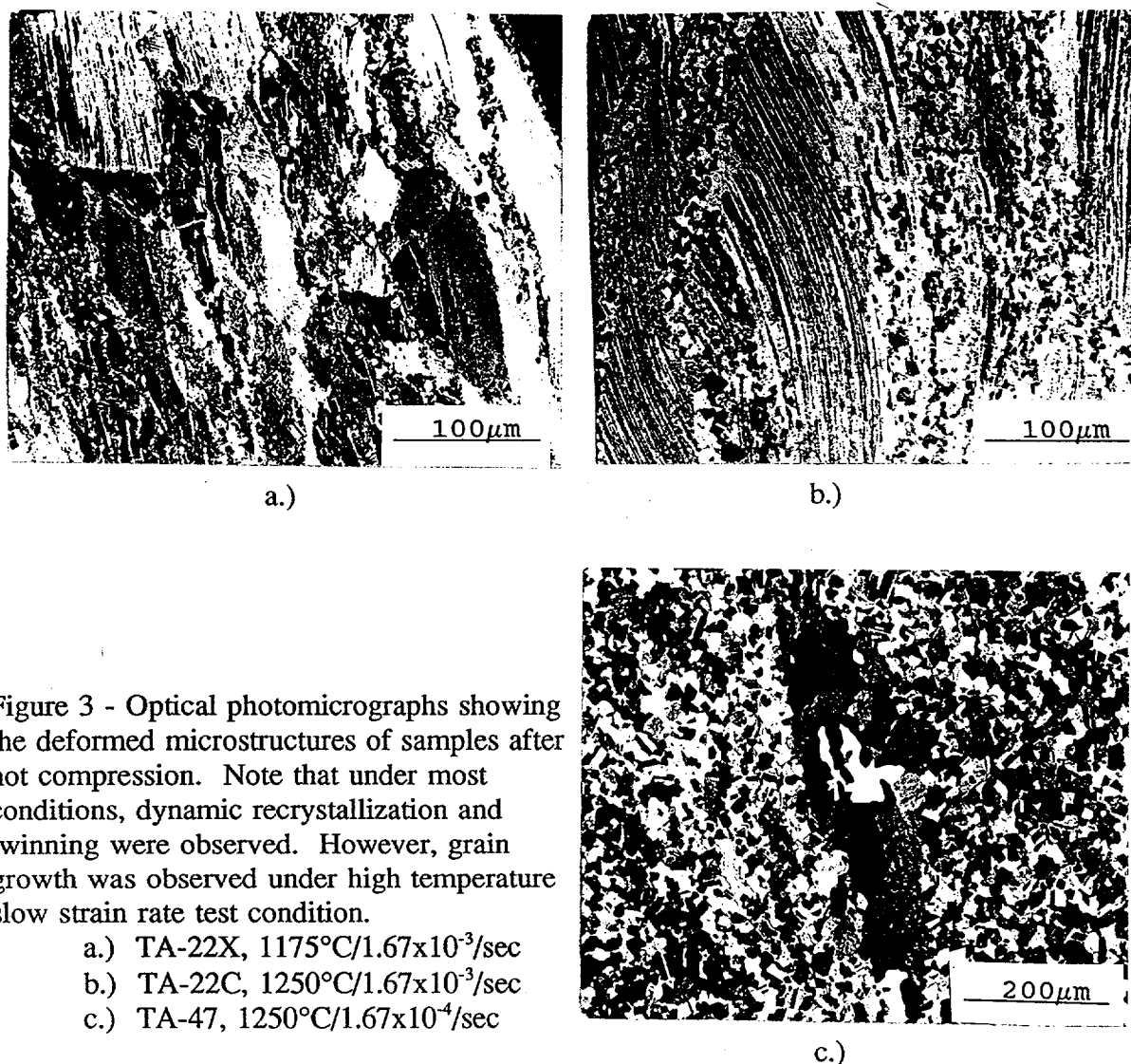


Figure 3 - Optical photomicrographs showing the deformed microstructures of samples after hot compression. Note that under most conditions, dynamic recrystallization and twinning were observed. However, grain growth was observed under high temperature slow strain rate test condition.

- a.) TA-22X, 1175°C/1.67x10<sup>-3</sup>/sec
- b.) TA-22C, 1250°C/1.67x10<sup>-3</sup>/sec
- c.) TA-47, 1250°C/1.67x10<sup>-4</sup>/sec

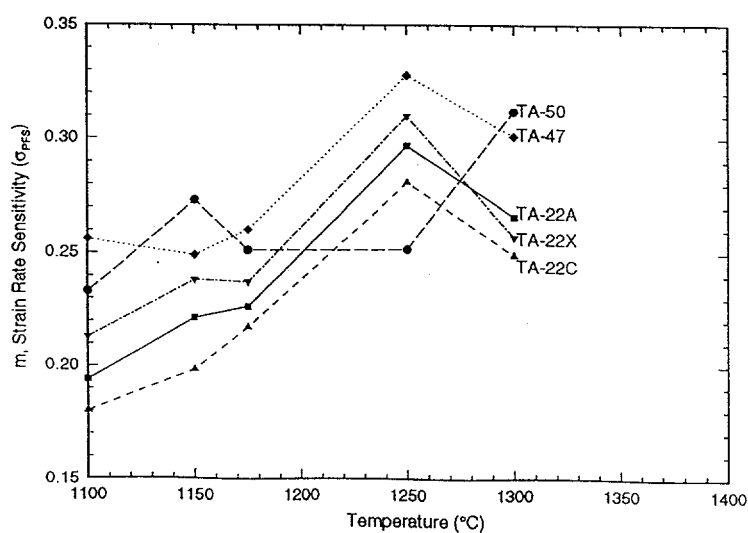


Figure 4 - The effect of temperature and processing on the strain rate sensitivity ( $m$ ) of Ti-48Al-2Nb-2Cr samples determined from the peak flow stress ( $\sigma_{PFS}$ ).