METALLOGRAPHY OF A HIGH STRENGTH MODIFIED 718 ALLOY - PWA 1472

J. F. Radavich

School of Materials Engineering

Purdue University

West Lafayette, IN 47907

Abstract

An electrolytic preparation technique for a high strength modified 718 alloy called PWA 1472 is described and used to structurally characterize various heat treated samples. As in alloy 718, the phases found are γ, γ'', δ, MC, and Laves.

Superalloys 718, 625 and Various Derivatives
Edited by Edward A. Loria
The Minerals, Metals & Materials Society, 1991

Introduction

Metallography has long been used as a basic tool for understanding and correlating structure and mechanical behavior of an alloy. As new superalloys are developed or old alloys modified, the standard procedure for determining the changes of structure and their effects on mechanical properties is by means of SEM or TEM techniques. The development of a family of superalloys which contain Nb as the primary carbide former and as one of the principal elements in the many strengthening phases has resulted in metallographic preparation problems when trying to achieve optimum metallography for these alloys. This problem is even more acute when trying to optimize the structures of cast versus wrought versions of the same alloy.

The development of such alloys as 718 or modified alloy 718 called PWA 1472 make use of the Nb rich phases to either control the grain size during processing or to strengthen the material through multiple heat treatments which produce delta, γ', or γ'' phases. The presence or absence of these phases as a result of the processing or aging treatments causes the matrix of the material to respond differently during metallographic preparations. In many cases the use of a mechanical polish and immersion etch technique yields inconsistent results.

The purpose of this paper is to describe an electrolytic preparation technique which can be used to characterize the phases found in samples of cast PWA 1472 which were given various thermal treatments.

Physical Metallurgy of PWA 1472

Alloy 1472 is very similar in composition to alloy 718 except the Ti is increased to 2%, the Cr is decreased to 12%, and the Nb is raised to 6%. The lower Cr level is designed to decrease the segregation effect of Cr to allow higher Nb levels without increasing the overall segregation behavior of the alloy. Since the Nb content determines the amount of strengthening phases, PWA 1472 should show higher strength levels after heat treatment.

The basic phases in cast 1472 should be similar to those found in alloy 718 -- namely MC, γ, γ', δ, and Laves. The temperature ranges in which these phase form and co-exist may be altered by the chemistry modifications. Because PWA 1472 is currently used as a cast alloy, the degree of segregation and the grain size requirements would determine the nature of the thermal treatments imposed on the material prior to aging.

The standard heat treatments for PWA 1472 are as follows:

	Coarse Grain	**Fine Grain**
Pre-HIP Homogenization:	2100°F (4) + 2125°F (4)	None
HIP:	2175°F/15 ksi/4 hours	2050°F/15 ksi/4 hours to 2175°F
Stabilization:	1600°F (1)	Same
Solution:	1850°F (1) Air cool or faster 100°F/hr	Same
Age:	1400°F (4) Cool 100°F/h 1225°F/2)	Same

It is apparent from the heat treatment that HIP closure of casting porosity and the homogenization of segregation are of prime interest as they would affect mechanical properties and structural responses.

Metallographic Preparation

The addition of more Nb should precipitate more γ and γ'' which are the strengthening phases in alloy 718 as well as in 1472. As the size of the strengthening particles becomes smaller, it is important that the flow layer from the last grinding operation be removed prior to etching. The total removal of the flow layer cannot be determined by an optical evaluation, but the high resolutions of the SEM or TEM will quickly show such artifacts. The key to produce a consistently good surface free of flow layer is by the use of electropolishing.

Electropolishing

A standard electropolishing electrolyte for 1472 is a 20% H_2SO_4 in methanol solution. The alcohol solution is kept cool while adding the acid and the solution is kept cool during electropolishing. The apparatus for electropolishing requires a DC power supply, a stainless steel beaker which is the cathode, and stainless steel crucible tongs to hold the sample which is the anode. After a 6 micron or finer diamond polish, the sample is electropolished for about 20 seconds at 30 volts and 2-4 amps. While electropolishing is taking place, the sample is continuously moved in order to bring fresh solution against the surface. After two alcohol rinses, the sample is dried and examined on an optical microscope.

The structural response to electropolishing varies with the phases present. The γ', γ'', and delta phases remain in the same plane as the matrix, while the MC, borides, or nitrides stand in relief. An SEM evaluation of such an electropolished structure would not show contrast or resolution; therefore, the sample must be etched.

Etching

Etching of electropolished samples can be carried out by either immersion or by an electro-etch. Because of the differences in thermal histories of heat treated samples, immersion etching of electropolished surfaces can produce inconsistent results. Generally, electropolishing produces a passive surface layer which must be dissolved before etching can take place. More importantly, the etching solutions tend to preferentially etch out the phases in question and resultant structures are not suitable for SEM studies.

An electrolytic etch gives the most consistent results. An ideal electro-etch should put the γ', γ'', and delta phases in relief to produce optimum contrast in the SEM. A standard etching solution which fulfills this requirement and is routinely used in our laboratory is composed of 170 cc of H_3PO_4, 10 cc H_2SO_4 and 15 grams of CrO_3 (1).

The same apparatus is used for electroetching as was used for electropolishing except the voltage is reduced to 5 volts and the etching time is 5-10 seconds. The sample is rinsed twice in fresh alcohol, dried, and examined in the SEM.

Metallographic Results

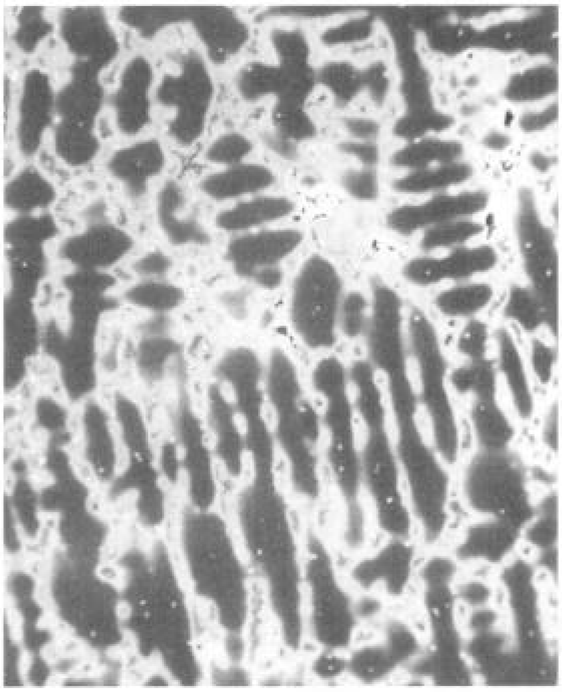
A number of 1472 samples representing some of the thermal conditions of the standard heat treatment for this alloy were selected for metallographic characterization. The following samples were electropolished and electro-etched as previously described:

1. As-cast large grain
2. As-cast fine grain
3. Pre HIP homogenization (2100°F/4h + 2125°F/4h)
4. Pre HIP homogenization + 1600°F/1h
5. Full heat treatment

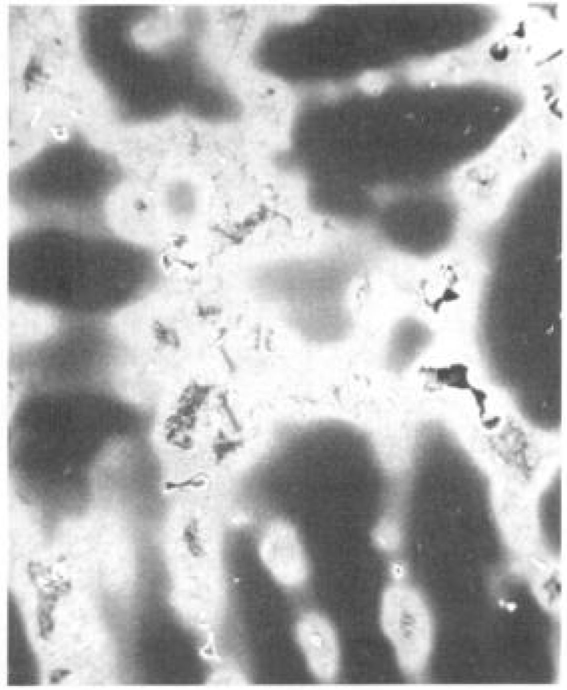
The SEM micrographs included in this paper were taken of representative structures after SEM evaluation of large areas.

Figure 1 shows a DAS pattern and representative structures in the large grain as-cast sample. The light regions are the interdendritic areas which have the highest segregation of Nb rich phases which precipitated on cooling. Within the interdendritic areas, MC, Laves, δ, γ', γ'', and porosity are concentrated. Due to a wide range of particle sizes, SEM magnifications of up to 30,000 X may have to be used to resolve the γ' and γ'' phases.

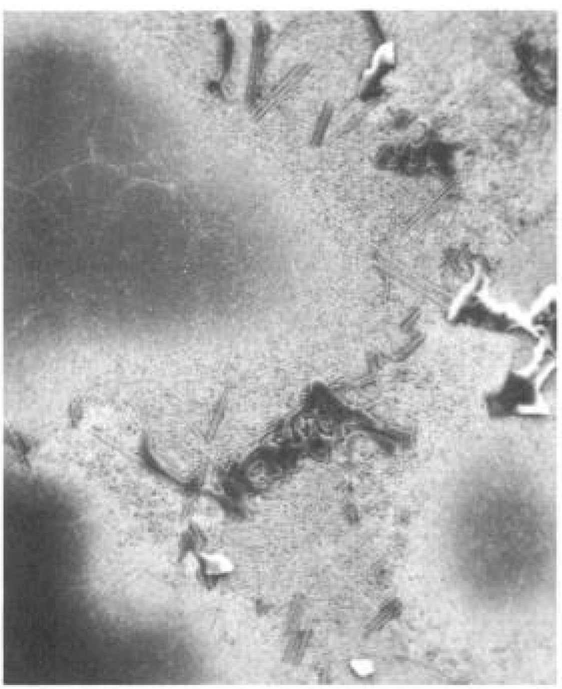
The electrolytic preparation causes the Laves phases to be flat and appear dark on the SEM, while the MC phase is in relief and appears bright. The white appearing areas



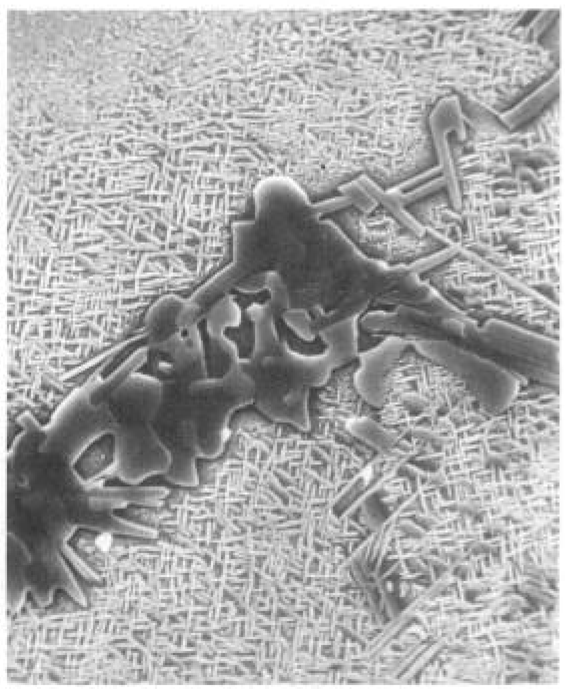
100X



300X

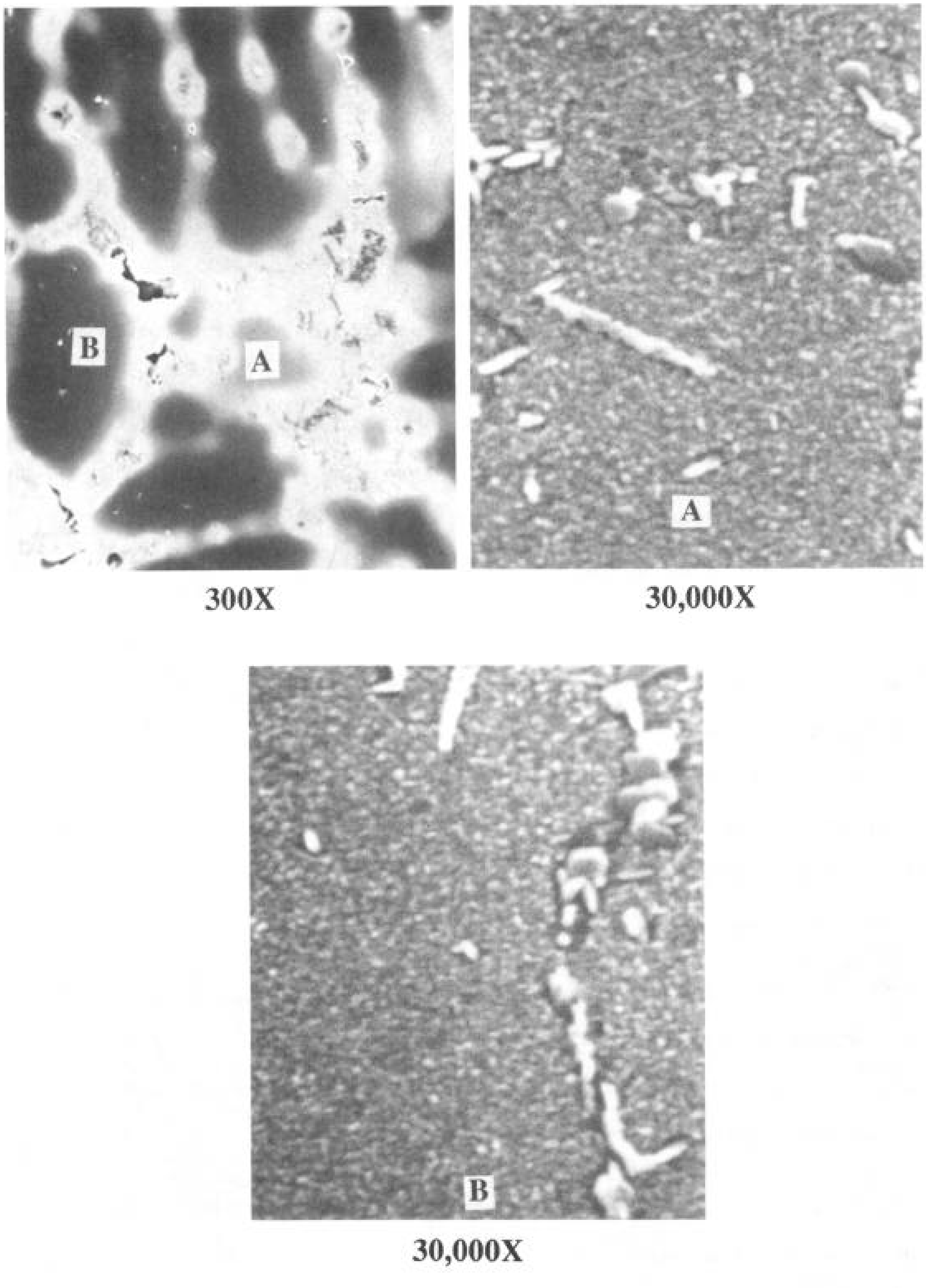


1000X



3000X

Fig. 1(a). Microstructure in As-Cast Large Grain PWA 1472.



300X 30,000X

30,000X

Fig. 1(b). Precipitation in Grey Area A and Dark Area B.

are a result of high Nb content which produce greater secondary electrons than the dark Nb lean areas.

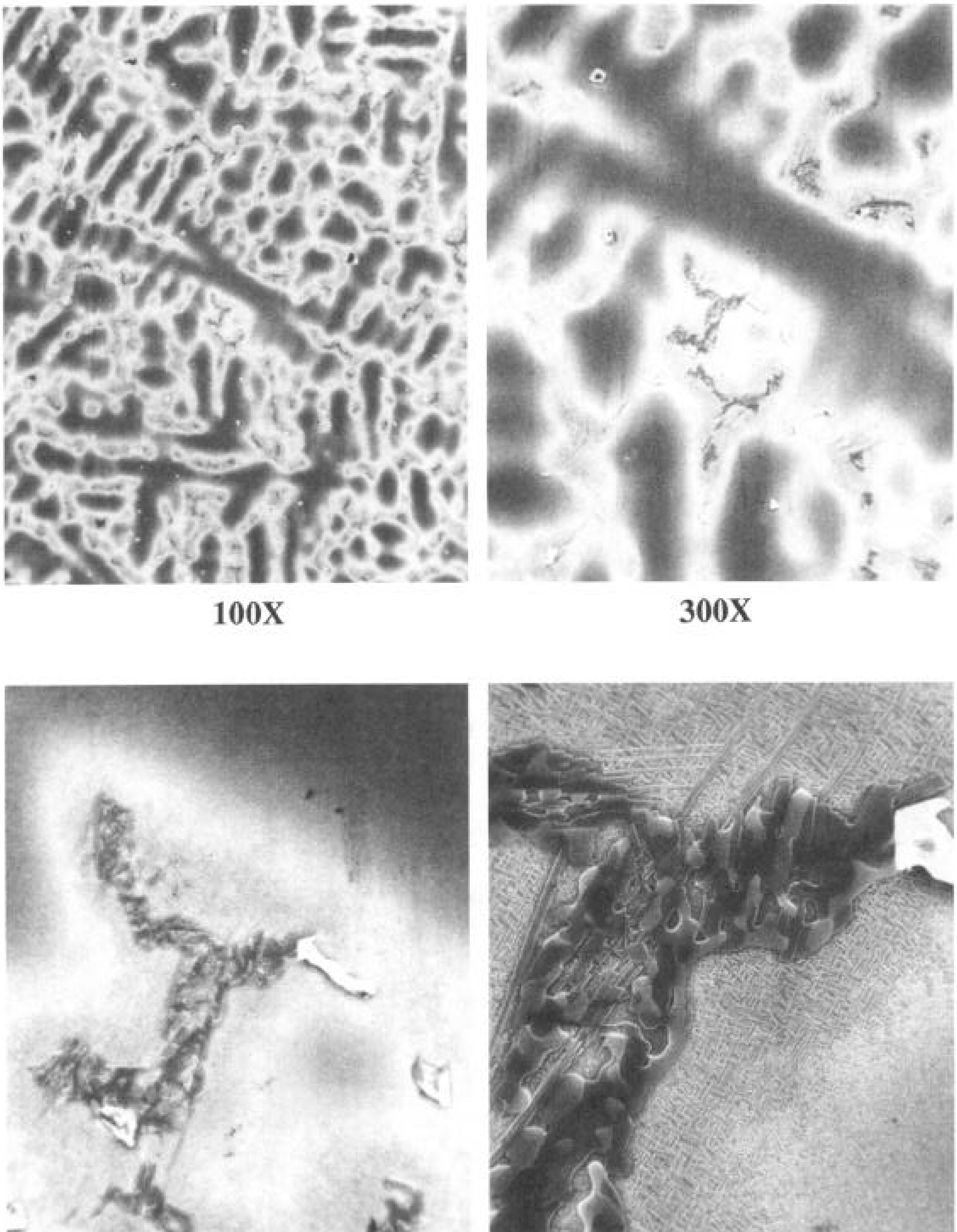
Figure 2 shows the smaller DAS structure in the fine grain as-cast sample. The same structures are present in the fine grain sample as were found in the large grain sample shown in Figure 1. The size of the phases is smaller and the degree of Nb segregation is much less.

Some degree of segregation is present after a Pre HIP homogenization cycle as seen in Figure 3. The γ'/γ'' precipitation in the white areas is very small since it formed during the rapid cooling of the material. It is evident that some Laves is still present and there appears to be more discrete MC particles in the segregated areas. Additional MC may be forming in the segregated areas due to the high Nb content and the high temperatures used for homogenization.

Figure 4 shows the effects of a "TAG" heat treatment used to determine the residual segregation present after the Pre HIP homogeneous heat treatment. When the Pre HIP sample is given a 1 hour age at 1600°F, γ'' should uniformly precipitate if no Nb segregation exists. However, evaluation at low magnifications show some residual segregation present as there are areas which appear darker than the surrounding matrix. In alloy 718, areas appear darker when the Nb is less than 4% and tend to precipitate round γ' rather than γ''.

When the dark areas in this sample are studied at high magnification, less γ'' precipitation and more spheroidal γ' particles are present. Similar studies of the white matrix areas show the opposite as more dense γ'' precipitation and very much less γ' precipitation are present.

Figure 5 shows the wide range of structures developed during the standard heat treatment. The delta phase is seen as plates as well as precipitates at the grain boundaries. As has been found in alloy 718, spheroidal γ' particles are present near grain



100X 300X

1000X 3000X

Fig. 2. Microstructure in As-Cast Fine Grain PWA 1472.

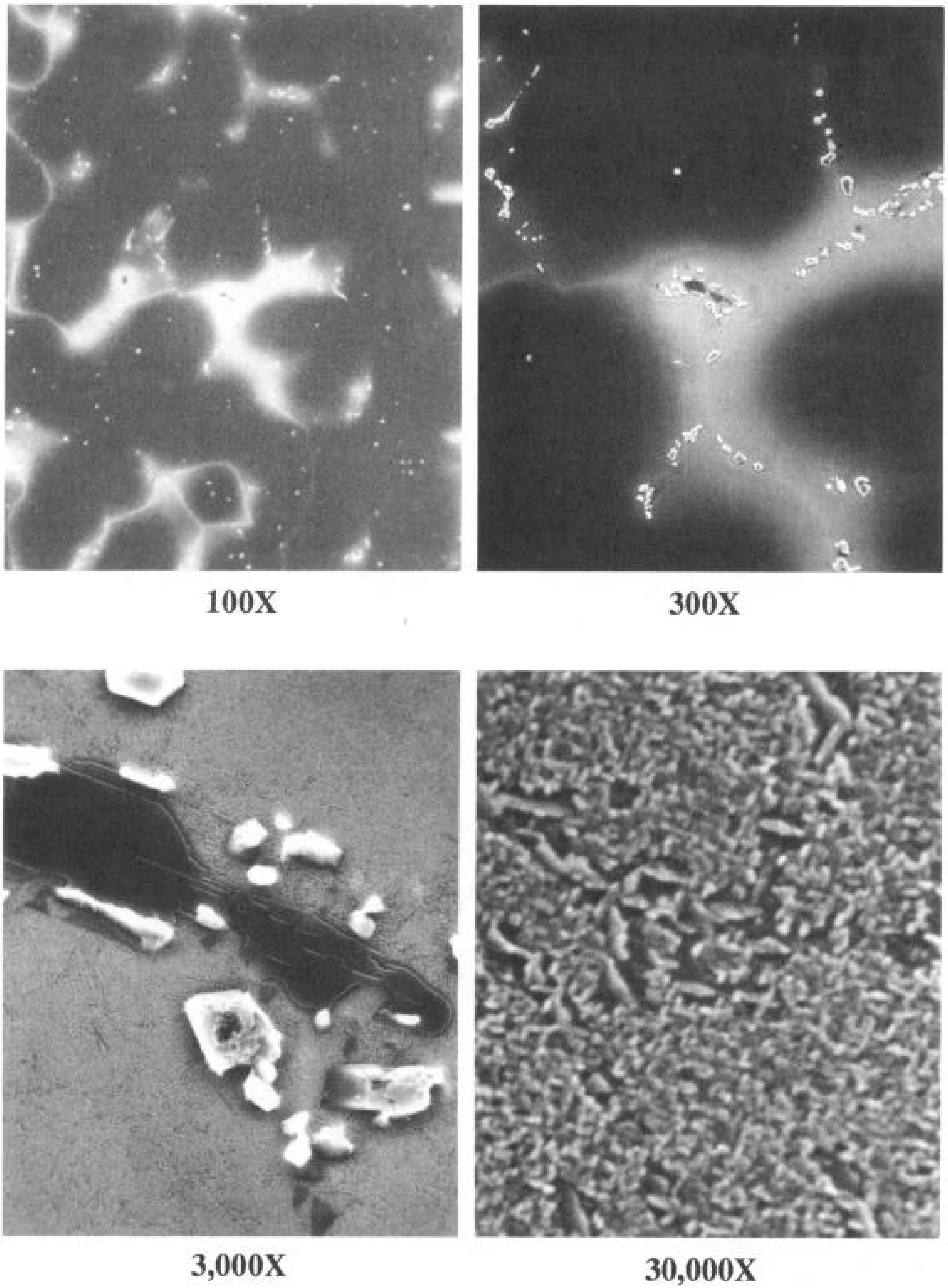
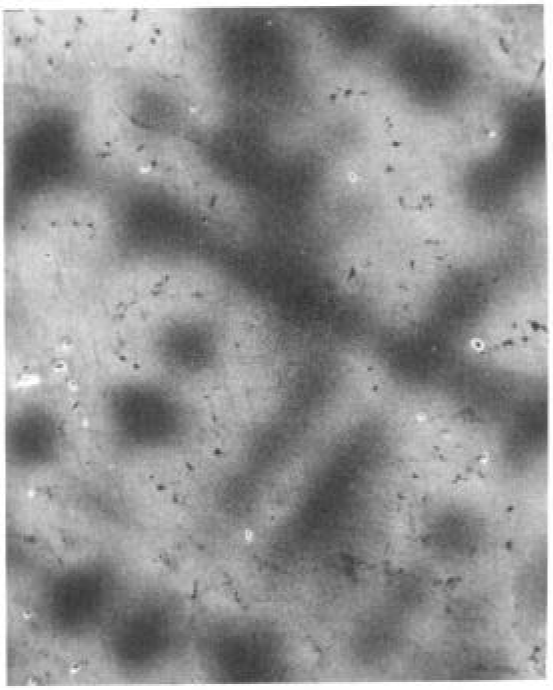
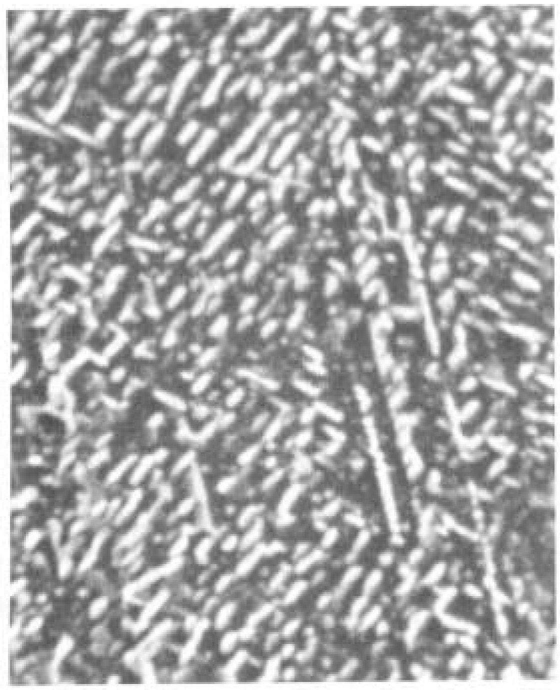


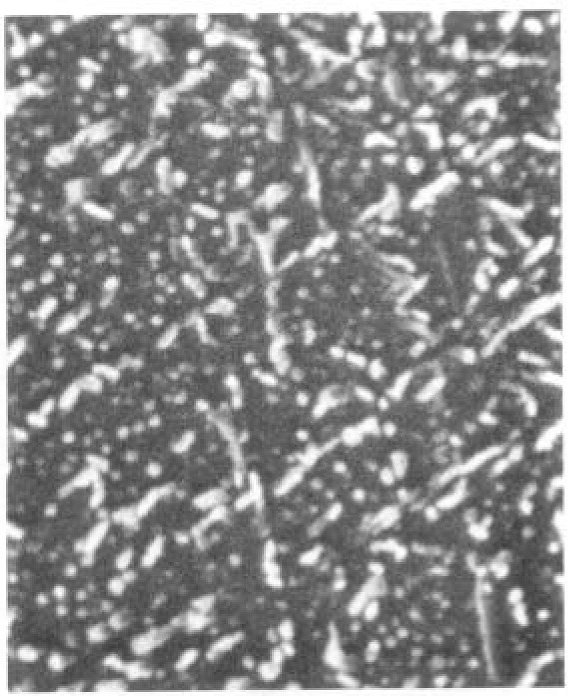
Fig. 3. Residual Segregation After Pre-HIP.



100X

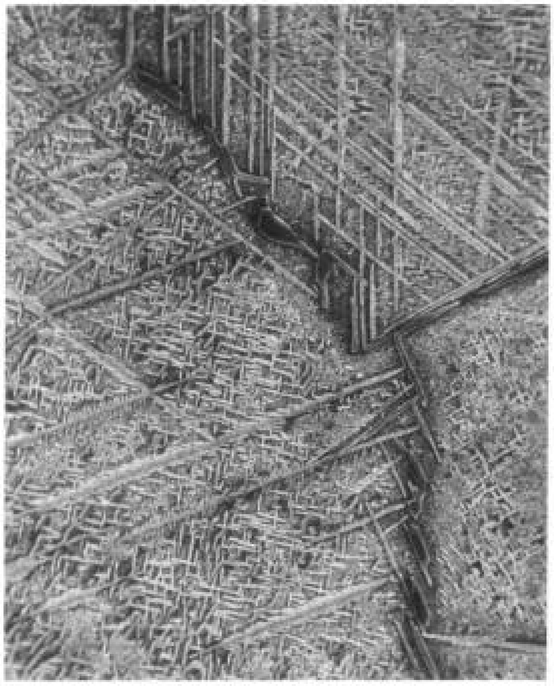


30,000X

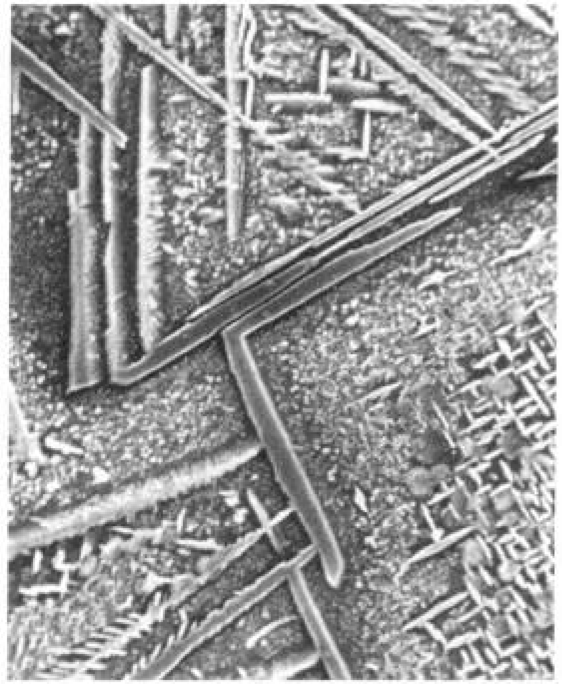


30,000X

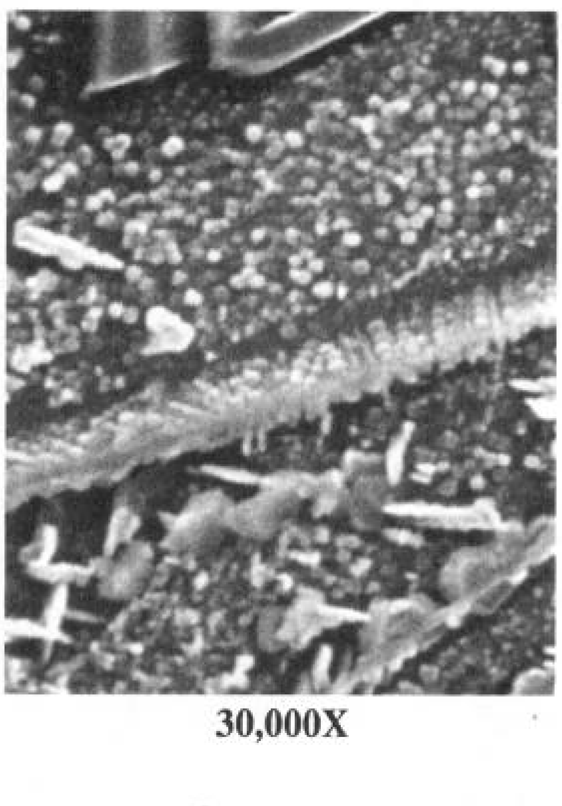
Fig. 4. Precipitation Behavior After Pre-HIP + 1600°F/1 hr.



3,000X



10,000X



30,000X

Fig. 5. Microstructure in Fully Heat Treated PWA 1472.

boundaries where extensive delta has formed at the expense of γ'. It appears that a transition of $\gamma' + \gamma \rightarrow \delta$ phase occurs in PWA 1472 similar to the transition in alloy 718.

Conclusion

The phases found in cast PWA 1472 are the same as those found in cast alloy 718-MC, Laves, γ, γ', and delta phase. The degree of segregation during solidification depends on the Nb content and the cooling rate. The precipitation of γ occurs when the Nb content in different areas is less than some nominal amount. In alloy 718, the γ precipitate is found when the Nb is less than 4%.

In PWA 1472, the electrolytic preparation technique provides a satisfactory method for revealing the phases in samples with different thermal histories.

Acknowledgements

The author would like to thank the financial support provided by the Niobium Products Co. The material for this study was provided by Pratt & Whitney Aircraft.

References

1. J. F. Radavich, "An Atlas of Superalloy Structures," to be published.