RAPID SOLIDIFICATION AND DYNAMIC COMPACTION OF Ni-BASE SUPERALLOY POWDERS

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Summary

A Ni-base superalloy containing 13%Al-9%Mo-2%Ta (in at.%) has been characterized in both the rapidly solidified condition and after dynamic compaction. Dynamically compacted specimens were examined in the as-compacted condition and observations related to current theories of interparticle bonding. In addition, the recrystallization behavior of the compacted material at relatively low temperature (~ 0.5 - $0.75\ T_m$) was investigated.

Introduction

During the recent past, there has been considerable work involving the rapid solidification processing (RSP) of Ni-base superalloys (1). Two of the advantages of rapid solidification have been exploited in these studies, namely increased homogeneity and refinement of microstructure. In general, the consolidation of superalloy particulate has been effected using conventional methods such as extrusion or hot isostatic pressing (HIP). These processes involve a prolonged thermal excursion where the temperatures approach that of the γ' solvus. In consequence, the resulting microstructures are somewhat coarser than that of rapidly solidified particulate and, in particular, the γ' distribution is on a scale comparable to that developed in material produced by conventional ingot metallurgy. It seems that processing with conventional consolidation techniques may cause much of the advantage associated with RSP to be lost, and it is felt that the approach of rapid solidification may only be exploited fully if compaction can be achieved in the absence of significant heating. Dynamic compaction appears to fulfill this requirement, and this paper describes work involving the application of this technique to the consolidation of rapidly solidified Ni-base superalloy particulate.

In the studies involving RSP of superalloy powders (1), much emphasis has been placed on the development of properties for turbine blade applications where components experience extremely high temperatures in service. In this case, the use of coventional consolidation techniques with the attendant thermal exposures may be understood, and the advantage of employing RSP is found to lie in the subsequent processing steps such as directional recrystallization (2). However, for applications where intermediate temperatures are appropriate, such as turbine disks, the use of conventional processing involving microstructural coarsening may offer significant disadvantages. This point may be illustrated by the following example. In a study involving the reduction of Cr from superalloys used in disk applications, one approach involves RSP and the partial substitution of certain other elements. In our study, RSP is being used in an attempt to improve the mechanical properties of these alloys by increasing the homogeneity of the microstructure. The microstructure typical of melt-spun ribbons of IN 713LC is shown in Fig. 1(a). To simulate the thermal exposure that is involved in the conventional consolidation of such particulate, ribbons were annealed for four hours at 1000°C and the resulting microstructure is shown in Fig. 1(b). It is clear that considerable coarsening has occurred; for example, large Cr-rich carbides have formed at intercellular boundaries. It is necessary, therefore, that compaction be effected without significant heating so that any microstructural decomposition be minimized.

In the work reported here, rapidly solidified powders of a Ni-base superalloy have been dynamically compacted to near theoretical density. The microstructures of the as-solidified particulate and the compacted samples have been characterized. Consolidated samples have been heat-treated over the temperature range 600-980°C and the microstructural response has been studied.

Experimental Procedure

The composition of the rapidly solidified powders used in this study was Ni-13Al-9Mo-2Ta (in at.%). The alloy was atomized using the RSR process (centrifugal atomization) and was kindly provided in powder form by Pratt & Whitney Aircraft, Government Products Division. Another form of particulate was produced by melt-spinning ribbons of the same alloy. These ribbons were reduced to particulate form using a method described elsewhere

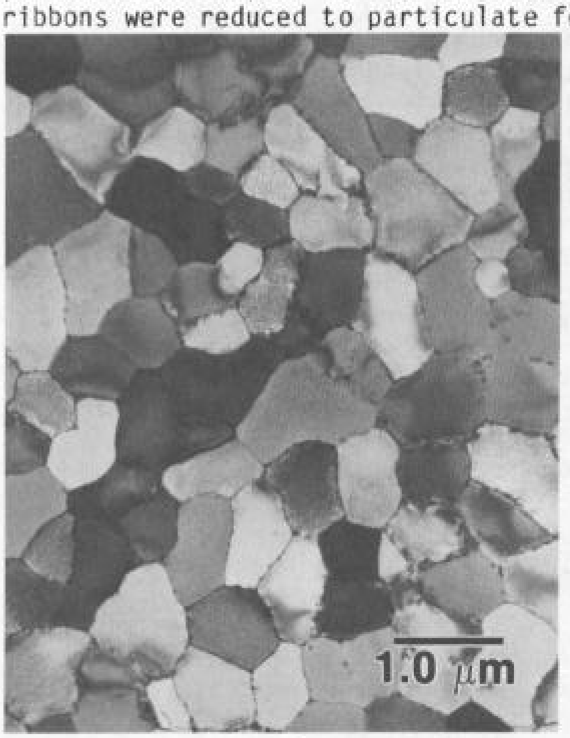

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Figure 1. Rapidly solidified IN 713LC ribbon a) as-solidified b) after 4 hours at 1000°C.

(3). Dynamic compaction was performed at Institut CERAC; consolidated samples in the form of right cylinders of ~5 cm in diameter and height were produced by causing a plastic projectile accelerated to a velocity of ~2000 ms^{-1} to be incident on the incipient compacts, in a manner described by Raybould, et al. (4). The microstructures of the rapidly solidified powders were characterized using analytical transmission electron microscopy (AEM) techniques, the thinned samples being prepared in a fashion described previously (5). The compacts were studied by both light optical metallography and AEM. In these studies, two electron microscopes were used, namely a Philips EM 400T and an EM 420, both operating at an accelerating voltage of 120kV.

Results and Discussion

Microstructures of Rapidly Solidified Particulate. The microstructures of RSR powders of the same composition as that used in the present study have been studied previously (6). It was found that most powders exhibited a refined dendritic microstructure with some elemental segregation being present in the interdendritic regions, as shown in Fig. 2(a). Some powders taken from the smaller sized particles (usually < 44 μm) possess microcrystalline morphologies, as shown in Fig. 2(b). There is little segregation associated with these types of microstructures and it has been proposed (7) that they form as a result of large degrees of undercooling being achieved prior to nucleation. The melt-spun ribbon consists of a cellular microstructure with a reduced amount of segregation, this being shown in Fig. 2(c). These various and contrasting microstructures exhibit three similar features when compared to conventionally cast and heat-treated materials, these being: i) a refined scale of structure (be that dendrite or cell spacings, or grain sizes), ii) reduced elemental segregation and iii) a distribution of γ' (the intermetallic compound based on Ni_3Al) the scale of which is in the range of ~20-80 Å. This latter observation may be illustrated by consideration of the superlattice dark-field electron micrograph shown in Fig. 2(d) which was taken from a powder particle exhibiting a dendritic morphology.

Microstructure of Compacted Material. The general features of the microstructure of compacted powders are shown in the optical micrograph in Fig. 3. It can be seen that many of the powders have been formed into an approximately polygonal shape as may be expected from the deformation associated with the propagation of the consolidating shock wave. The dendritic nature observed in many of the as-solidified powders is apparent in these parts of the microstructure. Other powders seem to have undergone marked changes in shape and have been forced to fill the interstices between the 'polygonized' powders. There are no recognizable microstructural features similar to those shown in Fig. 2. Morris has reported that there are regions of featureless contrast associated with the prior particle boundaries in optical micrographs of polished and etched samples of dynamically compacted superalloys (8). These regions, he claims, are to be considered consistent with a mechanism of consolidation which is thought to involve local melting and rapid solidification of the surface regions of particulate. In the present work, it was found that many variations in contrast could be produced depending on the etching conditions and it is concluded here that in the case of Ni-base material such observations are not useful in the determination of the compaction mechanism.

Transmission electron microscopy was employed to make observations of the microstructure of dynamically compacted samples and examples are shown

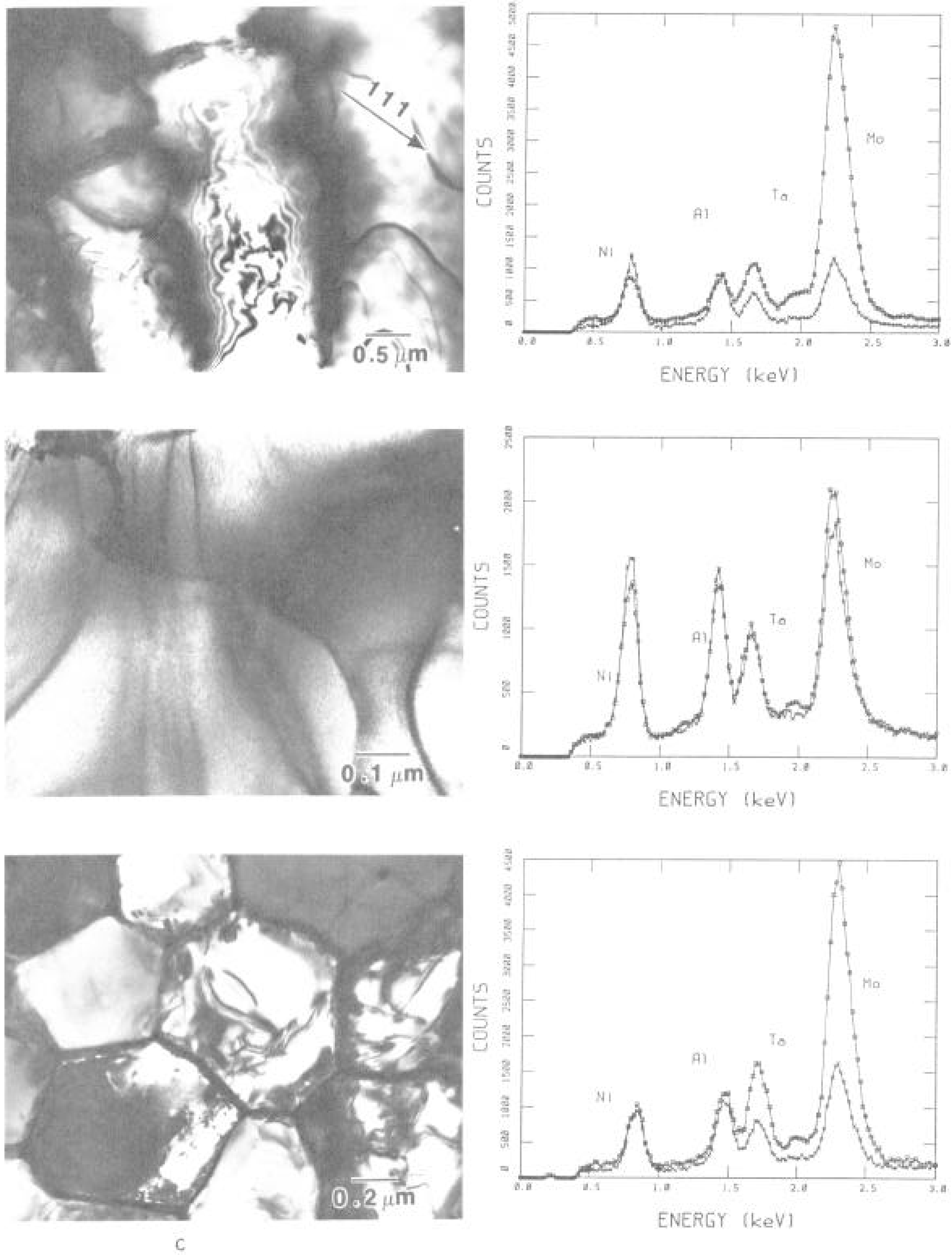

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Figure 2(a-c) Rapidly solidified Ni-base alloy (Ni-13Al-9Mo-2Ta) with associated X-ray spectra (taken in STEM mode). a) dendritic RSR powder with spectra from center of dendrite (X) and interdendritic precipitate (□) b) intersection of three grains in a microcrystalline RSR powder with spectra from center of grain (X) and grain boundary (□) c) melt-spun ribbon with spectra from center of cell (X) and intercellular precipitate (□).

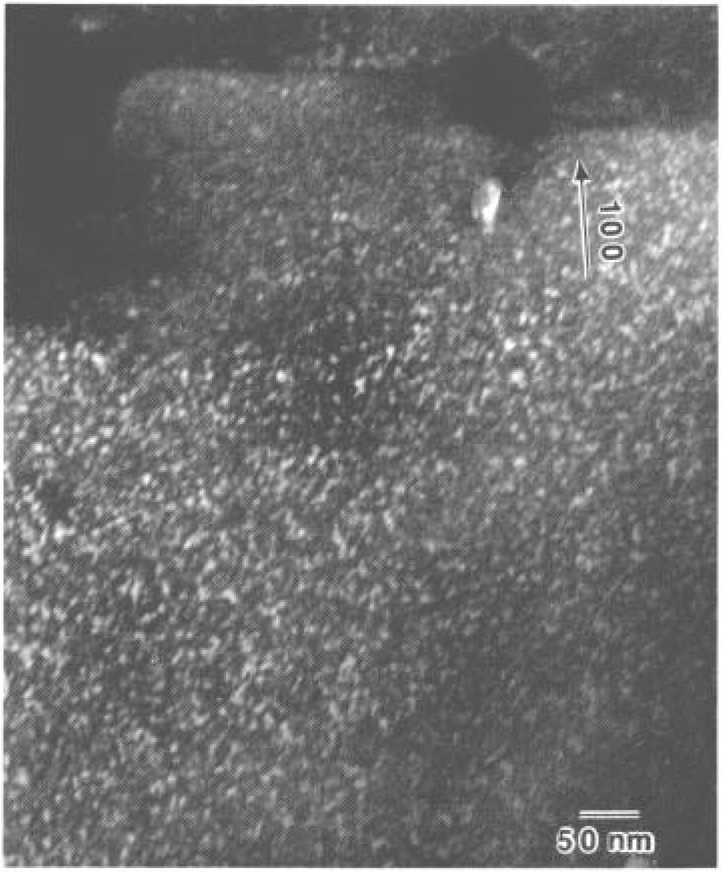

Figure 2d. Dark-field micrograph (taken from γ' superlattice reflection showing fine distribution of γ' in dendritic powder of figure 2a.

in Fig. 4. A typical region of a compacted piece is shown in Fig. 4(a); the microstructure is found to be heavily deformed, presumably due to the effects of the primary compacting shock wave and those caused by reflection. Of interest are the regions corresponding to the prior particle boundaries since, as mentioned above, the mechanism of compaction is thought to involve local surface melting. Such a region of a foil is shown in Fig. 4(b) and it is clear that the microstructure is significantly different from that shown in Fig. 4(a). It has been speculated (8) that these microstructural features correspond to melted regions, although more circumstantial evidence is required to substantiate this view. In the present work, a detailed diffraction study has shown that the elongated directions of the columnar grains present in Fig. 4(b) are parallel to <100>, the preferred growth direction of dendrites and cells in these alloys. This is consistent with the proposed mechanism of compaction but does not offer unequivocal proof.

Microstructures of Heat-Treated Samples. The microstructure of rapidly solidified Ni-base superalloy powders appears to be somewhat more refined than that of conventionally processed material. In addition, it seems that dynamic compaction does indeed result in consolidation of particulate without significant coarsening of the microstructure. Since it was intended that this processing approach be directed towards an application involving turbine disks, samples were heat-treated in the temperature range 760°-980°C for various times so that the response of the microstructure might be determined. It was found that at all temperatures the samples recrystallized. This is, at first, a rather surprising result since in order to recrystallize conventionally processed materials, it is necessary to anneal samples at temperatures approaching the γ' solvus. In view of this result, annealing at even lower temperatures was performed and it was found that samples would recrystallize when heated at 600°C. The reason

Figure 3. Optical micrograph of dynamically compacted Ni-base alloy (Ni-13Al-9Mo-2Ta) powders.

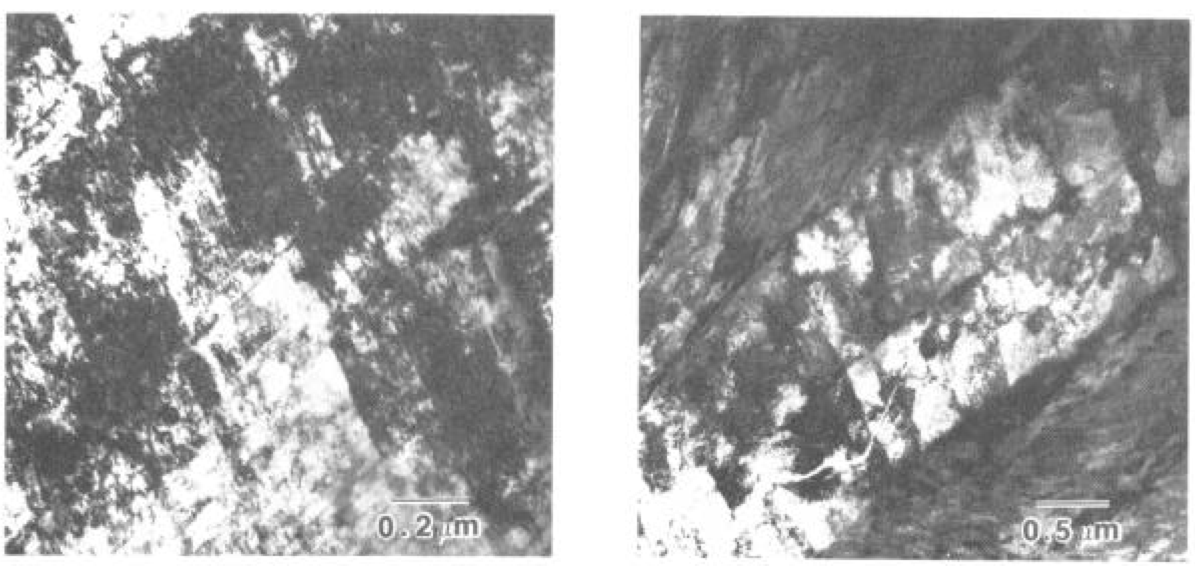

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Figure 4. TEM micrographs of as-compacted powders shown in figure 3 a) typical intraparticle region b) region showing possible example of interparticle melting.

for this relatively low temperature of transformation is thought to involve two factors. Firstly, there is an extremely large dislocation density present in compacted samples (Fig. 4). These defects would provide a large driving force for recrystallization. Secondly, the size of the γ' particles in the rapidly solidified powders is extremely small (Fig. 2(d)). In conventionally processed materials, where the γ' distribution is usually well developed, annealing takes place near the solvus so that particles may be dissolved and no longer act in a way to inhibit the motion of dislocations and grain boundaries, etc. In the present case, the γ' is so small that it does not, in all probability, act as an efficient barrier to dislocation motion and boundary migration. Thus, recrystallization may proceed at temperatures considerably lower than those employed normally.

A phase transformation occurs in alloys of the type used in the present study which may offer advantages in terms of disk applications. Thus, at temperatures $\sim$800°C, there exists the Ni_3Mo solvus. For this reason, it is of interest to consider the results of recrystallization performed at temperatures either above (980°C) or below (760°C) the Ni_3Mo solvus. Fig. 5 shows a recrystallization front in a specimen which was heat-treated for 5 hours at 980°C. By comparing the micrograph with the accompanying sketch, it may be noted that the recrystallized grain size is relatively fine ($\sim$0.5-1.0 μm) with large regions of γ'. Selected area diffraction (SAD) patterns of the recrystallized region (Fig. 6) reveal reflections from γ, γ', and a Ni_xMo intermetallic which is either Ni_3Mo (DO_{22}) or the (1 1/2 0) short range order structure. Figure 7 is a dark-field micrograph (taken from a Ni_xMo reflection) showing the fine distribution of this phase which formed during cooling from the recrystallization temperature.

Apart from the fully recrystallized regions described above, another type of recrystallized structure is observed. Here, the structure is somewhat less deformed than the as-compacted microstructure and consists of fine equiaxed grains. It is tempting to speculate that this is caused by a primary (continuous) recrystallization reaction which may occur in the more heavily deformed regions either during heating to temperature or in the early stages of the annealing itself. Subsequent coarsening of the γ' would inhibit normal grain growth, leading to the formation of secondary or discontinuous recrystallization fronts yielding the microstructure shown in Fig. 5.

Specimens which were heat treated at 760°C display microstructures similar to those found in specimens annealed at the higher temperature. The main difference involves the nature of the Ni_xMo phases. Fig. 8 shows the microstructure of recrystallized material with an associated SAD pattern. Diffraction maxima originating from both Ni_3Mo and Ni_4Mo phases are observed; presumably, these phases formed during the annealing treatment.

Conclusions

The results of the present study may be summarized as follows.

a) The microstructures of a rapidly solidified Ni-base superalloy have been characterized. In the case of RSR powders and melt-spun ribbons the microstructure is refined and the elemental segregation significantly reduced compared to that of conventionally processed material. In particular, the γ' size is in the range 20-80 Å.

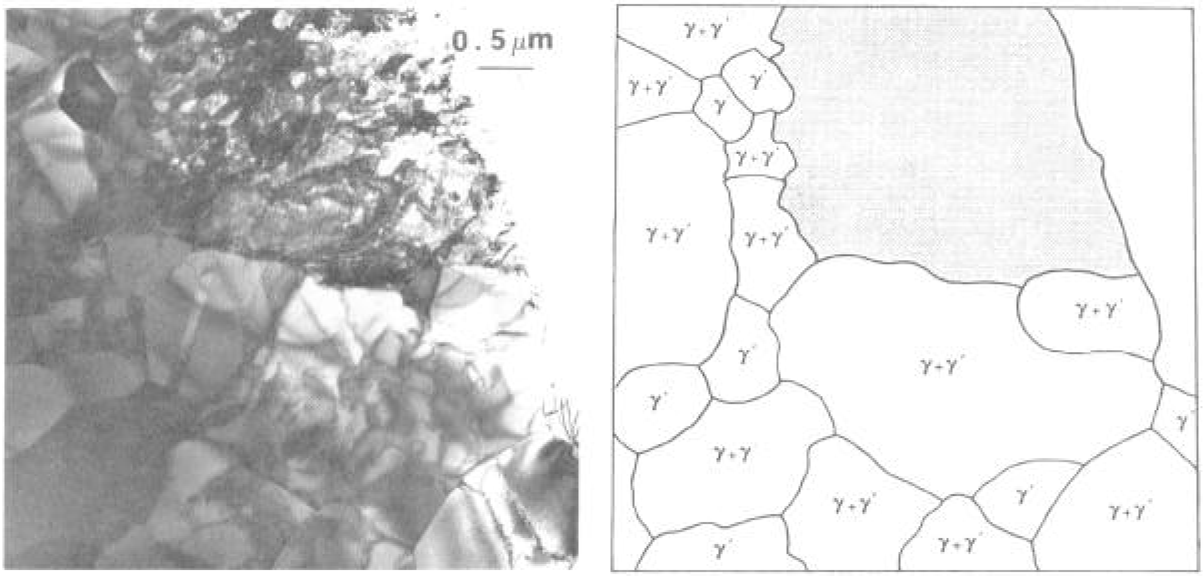
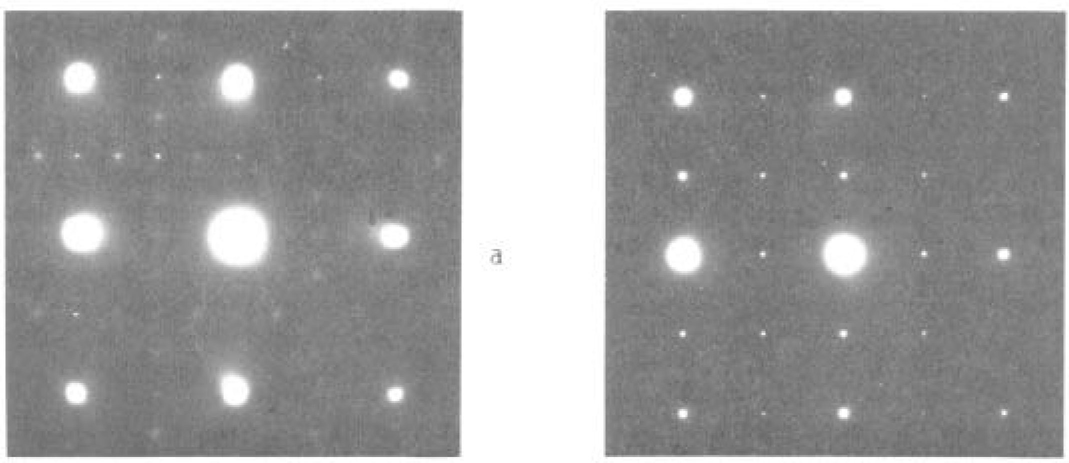


Figure 5. Recrystallization front in dynamically compacted (Ni-13Al-9Mo-2Ta) alloy after 5 hours at 980°C with schematic identifying nature of resulting grains.



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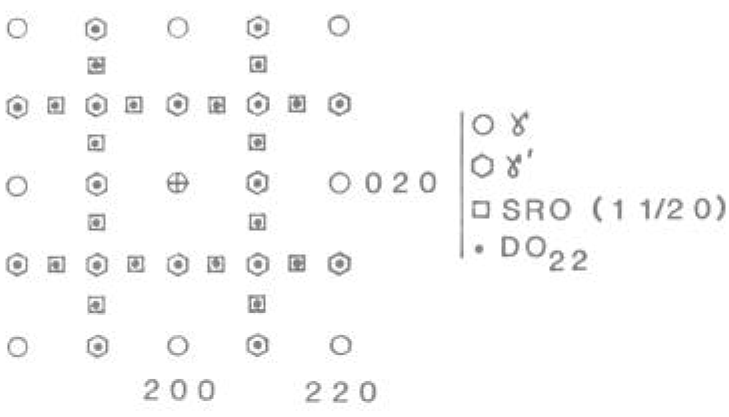

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Figure 6. Dark-field micrograph in fully recrystallized region shown in figure 5. a) from γ b) from γ' c) schematic showing reflections from various phases in the microstructures.

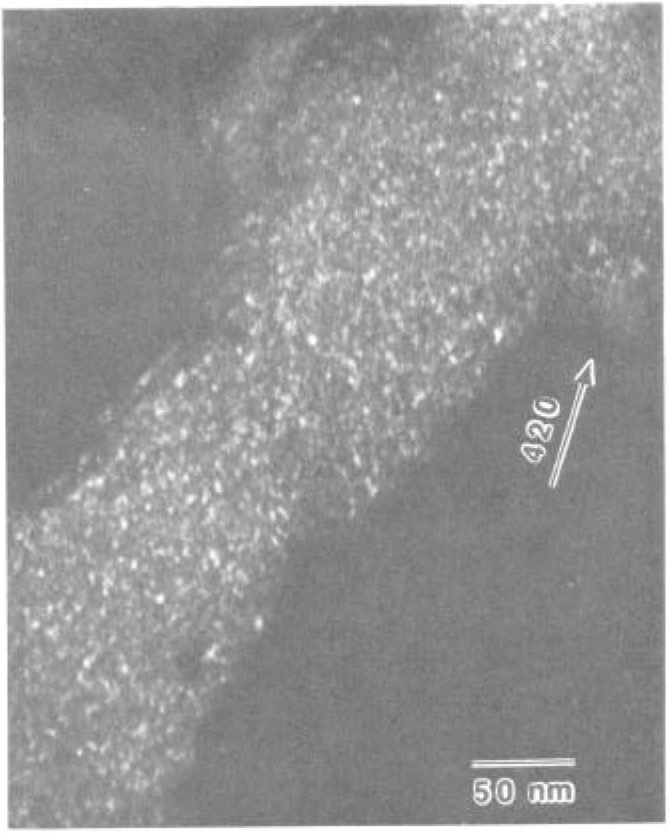

Figure 7. Dark-field micrograph in fully recrystallized region of figure 5 (taken from Ni_xMo reflection) showing fine distribution of the Ni-Mo intermetallic phase.

b) Dynamic compaction was employed to consolidate rapidly solidified powders of the alloy under study. There is some circumstantial evidence which suggests that in some locations of the sample, melting of prior particle boundaries has occurred, although this has by no means been established.

c) Heat-treatment in the temperature range 600°-980°C results in recrystallization of the samples. This behavior has been attributed to a large driving force from the presence of a high dislocation density in the compacted samples and the extremely refined distribution of γ'. The presence of the Ni_3Mo solvus at ~800°C leads to some subtle microstructural differences in samples which are recrystallized either above or below this temperature.

Acknowledgements

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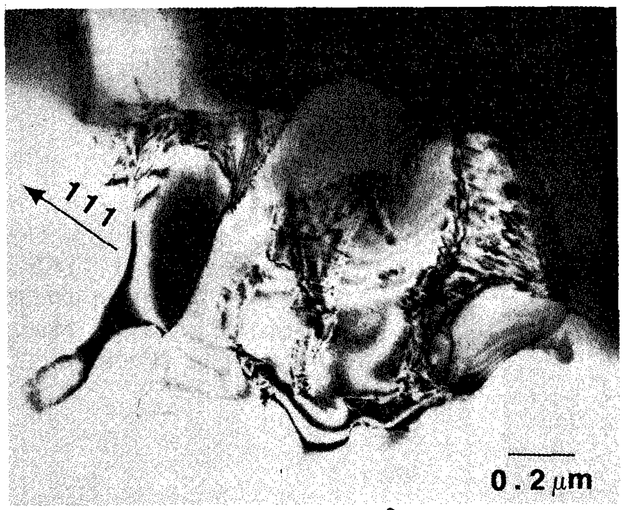

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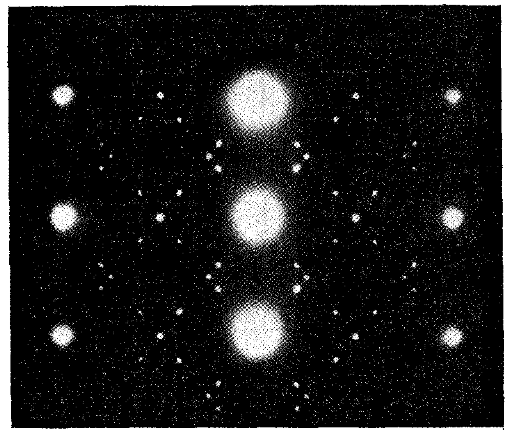

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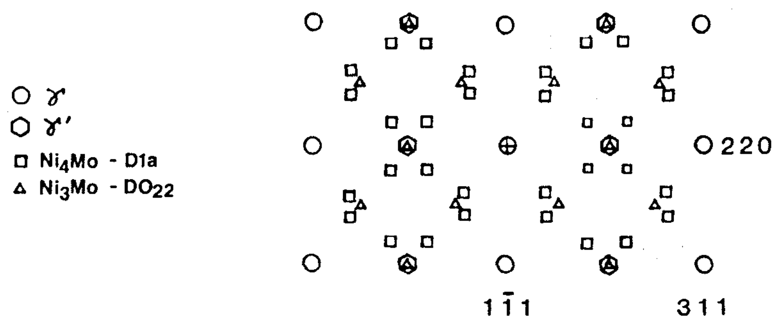
Figure 8. TEM micrograph and associated SAD patterns from fully recrystallized region of dynamically compacted (Ni-13Al-9Mo-2Ta) alloy alloy after annealing at 760°C. a) Bright field micrograph b) SAD pattern c) Schematic showing reflection from various phases in the microstructure.



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