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RESISTANCE OF A $\gamma/\gamma' - \delta$ DIRECTIONALLY SOLIDIFIED EUTECTIC ALLOY TO RECRYSTALLIZATION

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16. Abstract

The lamellar directionally solidified nickel-base eutectic alloy γ/γ' - δ has potential as an advanced turbine blade material. The microstructural stability of this alloy was investigated. Specimens were plastically deformed by uniform compression or Brinell indentation, then annealed between 705° and 1120° C. Microstructural changes observed after annealing included γ' coarsening, pinch-off and spheroidization of δ lamellae, and the appearance of an unidentified blocky phase in surface layers. All but the first of these was localized in severely deformed regions, suggesting that microstructural instability will not be a serious problem in the use of this alloy.

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RESISTANCE OF A γ/γ' - δ DIRECTIONALLY SOLIDIFIED EUTECTIC ALLOY TO RECRYSTALLIZATION*

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SUMMARY

A recurring concern about the use of directionally solidified eutectic alloys is the possible instability of plastically deformed eutectic microstructure on exposure to use conditions. This report presents the results of an investigation of microstructural stability of a γ/γ' - δ eutectic alloy of the nominal composition nickel - 20 percent columbium - 6 percent chromium - 2.5 percent aluminum (by weight). The effects of two modes of plastic deformation were investigated: (1) uniform compressive deformation up to 5 percent and (2) severe local deformation by Brinell indentation. Annealing treatments of up to 300 hours at temperatures from 705° to 1120° C were used following the deformations.

Uniform compressive deformation of 1 to 5 percent resulted in some surface and internal microcracks, usually following grain boundaries. Microhardness increased with plastic deformation, but the increase was essentially annealed out during a 1-hour anneal at 1100° C. Gamma prime coarsening was observed following 100-hour, 1100° C anneals of both as-cast and uniformly deformed specimens. The annealed γ' precipitate size appeared to decrease with increasing plastic deformation.

Specimens with the Brinell indentation load axis normal to the alloy growth direction experienced bending of lamellae to a depth of about 2 millimeters. Twinning of the δ lamellae was prevalent in these severely deformed regions. Annealing at 705° C resulted only in slight γ' coarsening. Annealing at 1120° C, in addition to δ spheroidization, resulted in a γ' depleted surface region, presumably due to oxidation effects. Annealing at 1040° C, in addition to a tendency for δ pinch-off, resulted in the formation of a blocky precipitate in the severely deformed region surrounding the Brinell indentation. When the indentation load axis was parallel to the alloy growth direction, severe lamellar splitting resulted. Subsequent annealing resulted only in γ' coarsening.

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INTRODUCTION

Directionally solidified eutectic alloys are of interest for application in advanced gas turbine engines. They offer a potential increase of 50° to 100° C in use temperature over commercial alloys now being used as turbine blades (ref. 1). One directionally solidified eutectic alloy currently under investigation for possible application as a future turbine blade material is the γ/γ' - δ alloy of nominal composition nickel - 20-weight-percent columbium - 6-weight-percent chromium - 2.5-weight-percent aluminum (Ni-20Cb-6Cr-2.5Al). Its microstructure consists of alternate lamellae of γ nickel solid solution and δNi_3Cb phase. The γ phase contains γ' Ni_3Al precipitates. The alloy composition was the most promising of those investigated by United Aircraft Technologies Laboratory (ref. 2).

One concern about these alloys has been the question of their microstructural stability over long periods at anticipated use temperatures. For example, could plastic deformation during processing, fabrication, installation, and operation (foreign object damage) of turbine blades eventually result in changes in microstructure (especially recrystallization or formation of embrittling phases) in critical regions, thereby degrading mechanical properties and reducing useful service life? One such investigation of a γ' - δ eutectic and a γ - γ' - δ ternary eutectic using Rockwell C indentation followed by high-temperature annealing, and using high-temperature ballistic impact, showed locally severe microstructural damage to the lamellae in some cases (ref. 3).

The study described herein was designed to determine whether this concern about microstructural instability was justified for the γ/γ' - δ composition examined. This alloy was deformed uniformly in compression to various degrees. It was also severely deformed locally with a Brinell hardness indenter. These deformations were carried out with the load axis both parallel and perpendicular to the eutectic growth direction. After deformation the material was subjected to annealing cycles in the 705° to 1120° C range for up to 300 hours and examined metallographically. This report describes the results of these tests and the conclusions which are drawn from them.

MATERIALS, APPARATUS AND PROCEDURE

Materials

Directionally solidified bars of the γ/γ' - δ eutectic Ni-20Cb-6Cr-2.5Al, 1.2 centimeters in diameter and 7.6 centimeters long were produced by United Aircraft Technologies Laboratory. A solidification rate of 3 centimeters per hour was used in a modified Bridgman furnace having a temperature gradient of at least 200° C per centimeter (ref. 2). The specimens used in this research were machined from these cast bars.

Uniform Compressive Plastic Deformation

To study the effects of known amounts of plastic deformation and of subsequent annealing on the behavior of $\gamma/\gamma' - \delta$ microstructure, specimens were given uniform compressive plastic deformations of 1, 3, and 5 percent at room temperature (strain rate 0.07/min). For 0.8-centimeter-long, 1.0-centimeter-diameter cylindrical specimens the load axis was parallel to the growth direction and for 0.8 by 0.8 by 1.0 centimeter parallel-sided specimens the load axis was perpendicular to the growth direction. These specimens were then annealed at 1100° C for 1, 22, and 100 hours in flowing argon followed by an argon quench.

The change in hardness due to the plastic deformation and the subsequent anneals was investigated by microhardness measurements made on both the transverse (perpendicular to the alloy growth direction) and longitudinal (parallel to the growth direction) surfaces. Indentations were made with a Wilson Tukon Microhardness tester (500-gram load and a diamond pyramid indenter) on slightly etched surfaces in approximately similar areas with good lamellar alinement. Each indentation spanned about 15 γ/γ' and 15 δ lamellae.

Severe Local Plastic Deformation

To simulate severe local deformation (e.g., foreign object damage), specimens were plastically deformed at room temperature using a Brinell hardness tester with a 10 millimeter ball and 2000-kilogram load applied for approximately 10 seconds. Brinell indentations were made both on transverse surfaces (the end surfaces of 0.6-cm long and 1.2-cm-diam cylinders) and on longitudinal surfaces (two 0.6- by 0.7-cm flats ground on the sides of the 0.6-cm-long cylinder, parallel to the growth direction). The specimens were subsequently annealed at 705°, 1040°, and 1120° C for 30, 100, and 300 hours in a flowing argon atmosphere followed by an argon quench.

Metallography

Evidence of any microstructural instability for both the compressively deformed and indented specimens was sought primarily by light metallography. The specimens were polished using normal metallographic procedures. The final polish was obtained with 0.05-micrometer alumina on microcloth. Etching was performed by immersing the specimens for approximately 5 seconds in a solution of 30 cubic centimeters nitric acid, 30 cubic centimeters water, 30 cubic centimeters acetic acid, and 1 cubic centimeter hydrofluoric acid. Scanning electron microscopy was occasionally used for higher resolution and chemical analysis.

RESULTS AND DISCUSSION

Uniform Compressive Plastic Deformation

No evidence of gross microstructural instability was observed for the compressively deformed and subsequently annealed specimens. Figure 1 shows the effect of plastic deformation and the subsequent anneal. The micrographs shown in figure 1 are of transverse sections. The 5-percent plastic deformation with the load axis perpendicular to the growth direction did not result in any significant change in the general appearance of the structure (fig. 1(b)). A small amount of cracking of the δ lamellae, due to the imposed deformation (fig. 1(b)), was observed. No delta twinning was observed. The subsequent 100-hour anneal at 1100° C did not produce any structural change other than γ' coarsening (fig. 1(c)). The γ' particle size, however, appeared to decrease with increasing amounts of prior plastic deformation (compare figs. 1(c) and (d)). This effect was also observed for γ/γ' - δ specimens compressively deformed 1, 3, and 5 percent with the load axis parallel to the growth direction (fig. 2). This effect of prior deformation on γ' particle size after annealing has not yet been satisfactorily explained.

Microcracks were observed in all the specimens that had been uniformly plastically deformed, more in the specimens deformed with the load axis perpendicular to the growth direction (fig. 3) than in those deformed with load axis parallel to the growth direction. The cracks were observed both at the outer lateral surface (fig. 3(b)) and inside the material (fig. 3(a)). However, the cracking was more pronounced at the lateral surface. The cracks usually followed grain boundaries.

Figure 4(a) shows the change in microhardness as a function of the amount of plastic deformation (load axis perpendicular to the growth direction) before annealing. As expected, the microhardness increased with the plastic deformation. However, higher hardness values (about 30 DPN higher) were obtained on transverse surfaces than on the longitudinal surfaces. On a transverse surface, with the microhardness load axis parallel to the growth direction, the hard δ lamellae would be loaded in compression as columns. However, on a longitudinal surface, the microhardness indenter would apply a bending load to the average δ lamellae. This would not be resisted as effectively, and the longitudinal surface hardness should be lower, as observed.

Even though the subsequent anneal of these plastically deformed specimens did not cause any recrystallization, the microhardness measurements indicate that, probably as a result of recovery, the hardness increase resulting from the plastic deformation is almost completely annealed out during the first hour of anneal at 1100° C (fig. 4(b)).

Severe Local Plastic Deformation

Micrographs obtained after plastic deformation and before annealing (fig. 5) show the effects of the deformation around and under the Brinell indentation. The indented surface after fine grinding, polishing, and etching is shown in figure 5(a). (The re-entrant curve of the edge at the top of fig. 5(a) actually corresponds to the periphery of the indentation. The indentation load axis, parallel to the growth direction, was normal to the plane of the micrograph). This micrograph shows that the ends of lamellae, which were parallel to and close to the outside edge of the indentation, bent to accommodate the severe plastic deformation. Those lamellae with ends perpendicular to and close to the indentation outer rim cracked to accommodate the deformation.

Figure 5(b) shows at higher magnification a longitudinal section through the indentation, where the load axis was parallel to the growth direction. Severe bending of the lamellae ends, which had been directly in contact with indenter surface, produced interlamellar splitting. Extensive bending of the lamellae under a Brinell indentation (up to a depth of about 2 mm) where the load axis was perpendicular to the growth direction may be observed in figure 5(c), which shows a longitudinal section through the indentation. Interlamellar splitting was occasionally observed, especially on the surfaces of the specimen parallel to the load axis (possibly due to the small sample size) (fig. 5(d)). However, few interior microcracks were noticed in regions below the indentation despite the severe lamellar bending. Extensive mechanical twinning in the δ plates in regions with the bent lamellae was observed in specimens indented with load axis perpendicular to the growth direction (fig. 5(e)). Cellular δ regions displayed heavier deformation twin density than the lamellar regions. The high amount of plastic deformation which the δ lamellae can accommodate without cracking is quite noteworthy. It is probably due to the δ twinning and the fact that δ lamellae are supported by the ductile γ. It also suggests that γ/γ' - δ could absorb a great deal of energy locally so that foreign object damage might be restricted to localized regions.

The effect of the 300-hour anneal at 1120° C after deformation is indicated in figure 6, which shows portions of a longitudinal section through the indentation where the load axis was perpendicular to the growth direction. The growth direction is in the plane of the micrographs. Close examination of figure 6(a) (see also figs. 6(b) and (c)) shows that some partial structural degradation has occurred in the severely plastically deformed region (bent lamellae region directly under the indentation). The regions to the side of the indentation do not show this degradation. This microstructural damage appears to involve localized pinching off of δ plates and a tendency for the resulting segments to spheroidize in the severely plastically deformed regions (fig. 6(c)). It did not occur in the regions that were not plastically deformed or that were only moderately deformed. Here only γ' coarsening was observed (fig. 6(b)).

The δ pinch-off and spheroidization is more clearly demonstrated in figure 6(d), which shows a longitudinal section through the heavily deformed region (directly under the indentation) after a 300-hour anneal at 1120^{O} C. It is suggested that the δ pinch-off starts at faults (twins) during the anneal and results in the observed partial spheroidization of the δ phase. Cellular δ regions appear to be more prone to such structural degradation than the lamellar δ regions. A γ' denuded zone can be observed near the surface in this micrograph, where some recrystallization of γ and δ can also be observed. In figure 6(a) the extent of γ' denuded zone at the alloy surface (up to a depth of about 100-μm, apparently due to oxygen present as a normal contaminant in the argon annealing furnace atmosphere) appears to be increased by the plastic deformation. The 30- and 100-hour anneals at 1120^{O} C also resulted in structural degradation of γ/γ' - δ, similar to that from the 300-hour anneal at this temperature, though to a lesser degree. No δ pinch-off was observed in specimens which were indented with the load axis parallel to the growth direction and annealed at 1120^{O} C; only γ' coarsening and a γ' denuded zone at the surface were observed.

The tendency for δ pinch-off can also be observed in the heavily deformed region after a 300-hour anneal at 1040^{O} C in figure 7, which shows the region immediately below the indentation where the load axis was perpendicular to the growth direction. However, for this anneal the structural degradation was much less severe than for the 1120^{O} C anneal and was confined to a much shallower region immediately below the indentation. A blocky precipitate can be observed near the surface up to a depth of about 40 micrometers. This phase appears from its morphology and etching characteristics to be γ', but compositional data (from energy dispersive X-ray spectrometry in the scanning electron microscope) are not yet adequate to confirm this. The presence and distribution of these precipitates is more clearly shown in figure 8, which shows the indented surface following a 300-hour anneal at 1040^{O} C after a light polish and etch to reveal the microstructure. The load axis was perpendicular to the growth direction. It may be noted that these blocky precipitates are localized near the indentation. The γ' bands have previously been observed to form in the area of impingement during high-temperature ballistic impact testing of the ternary γ - γ' - δ eutectic alloy (ref. 3). Even the plastic deformation caused by cutting γ/γ' - δ with a silicon-carbide cut-off wheel was occasionally severe enough to cause these precipitates to form at the cut alloy surface during a 100-hour anneal at 1040^{O} C.

Figure 9 shows that γ/γ' lamellae in the vicinity of the blocky precipitates contain much finer γ' particles than those away from blocky precipitates. This suggests that these blocky precipitates grew at the expense of γ' precipitates in the γ/γ' lamellae. Similar microstructural changes were observed in the Brinell-indented specimens (load axis perpendicular to the growth direction) following the 30- and 100-hour anneals at 1040° C, though to a much lesser extent.

The blocky phase was observed only after 1040° C anneals. Exposure at 1120° C resulted in the formation of denuded zone instead (compare figs. 10(a) and (b)). Low temperature annealing at 705° C, following the Brinell indentation, did not result in any microstructural change, except some slight γ' coarsening.

Figure 11 shows the effect of 30-hour anneals at 705°, 1040° and 1120° C on the deformation twins in the severely deformed δ regions (bent lamellae region due to the Brinell indentation). The 705° C anneal did not result in microstructural change in δ twins (fig. 11(a)). The 1040° C anneal resulted in precipitates, believed to the γ' at twins in the region immediately below the indentation (fig. 11(b)). The twin density was also observed to be less in the annealed than in the as-deformed specimens. Very few twins were observed after the 1120° C anneal, and the ones observed were wider than those in the as-deformed specimens (fig. 11(c)). This anneal resulted in delta pinch-off starting along the twin boundary (fig. 11(c)). It is postulated that grooving of the δ plates occurs along the twins thereby reducing the surface energy. Adjacent γ or γ' phases grow, following the advancing groove, eventually resulting in δ pinch-off.

CONCLUDING REMARKS

The γ/γ' - δ eutectic alloy investigated, when uniformly deformed up to 5 percent is not expected to exhibit gross microstructural instability during prolonged use at temperatures as high as 1040° C. Severe local plastic deformation followed by annealing for 30 to 300 hours at 1120° C produced some δ pinch-off and occasional δ spheroidization. However, the 1120° C temperature is well above the expected temperature of even moderately stressed sections of turbine blade airfoils. The blocky precipitate, formed in the severely plastically deformed regions after 100- to 300-hour anneals at 1040° C, was limited to localized surface layers and would not be expected to affect bulk mechanical properties. The excellent ability of δ lamellae to bend without cracking, especially during indentation on the longitudinal surfaces, also suggests the capability of the γ/γ' - δ eutectic alloy to localize foreign object damage. However, properties such as fatigue resistance or oxidation resistance, which are related to surface characteristics may be affected by such microstructural changes as were observed.

SUMMARY OF RESULTS

The microstructural stability of plastically deformed γ/γ' - δ directionally solidified eutectic alloy (Ni-20Cb-6Cr-2.5Al) was investigated. Two modes of plastic deformation were used: (a) Uniform compressive deformation up to 5 percent, and (b) severe local deformation by Brinell indentation. Annealing treatments of up to 300 hours at temperatures from 705° to 1120° C were applied after each deformation treatment. The following results were obtained from this study.

1. No evidence of microstructural instability or recrystallization, except γ' coarsening, was observed in specimens uniformly compressively deformed up to 5 percent and annealed at 1100° C for up to 100 hours. The γ' precipitate size following anneals at 1100° C appeared to decrease with the increasing amount of plastic deformation. Uniform compressive plastic deformation of 1 to 5 percent resulted in some surface and internal microcracks, usually following the grain boundaries.

2. The microhardness was observed to increase with increasing plastic deformation. Higher hardness values were obtained on transverse surfaces (normal to the alloy growth direction) than on the longitudinal surfaces. Hardness increase resulting from the plastic deformation was almost completely annealed out during the first hour of anneal at 1100° C.

3. Brinell indentation resulted in bending of lamellae to a depth of about 2 millimeter and some lamellar splitting.

4. In the severely deformed region (to a depth of about 2 mm) of the indented specimens (load axis perpendicular to the growth direction), pinching off of δ lamellae and their occasional spheroidization was observed following 30- to 300-hour anneals at the highest temperature, 1120° C. The tendency for such a δ pinch-off was also observed in similarly deformed regions after a 300-hour anneal at 1040° C. However, for this anneal it was much less severe and was confined to a much shallower region immediately below the indentation. It is believed that δ pinch-off started at twin boundaries and resulted in the subsequent delta spheroidization.

5. Annealing of indented specimens for 100 to 300 hours at 1040° C resulted in the formation of as-yet unidentified blocky precipitates, at the deformed surface (up to a 40 μm depth), especially near the Brinell indentation. The 30- to 300-hour anneal at 1120° C resulted in a γ' denuded zone at the specimen surface (up to a depth of about 100 μm). Precipitates, assumed to be γ', were observed to form along the twins in the severely deformed δ regions after anneals at 1040° C. Anneals at 705° C did not produce any microstructural change, except slight γ' coarsening.

Lewis Research Center,

National Aeronautics and Space Administration,

Cleveland, Ohio, July 11, 1975,

505-01.

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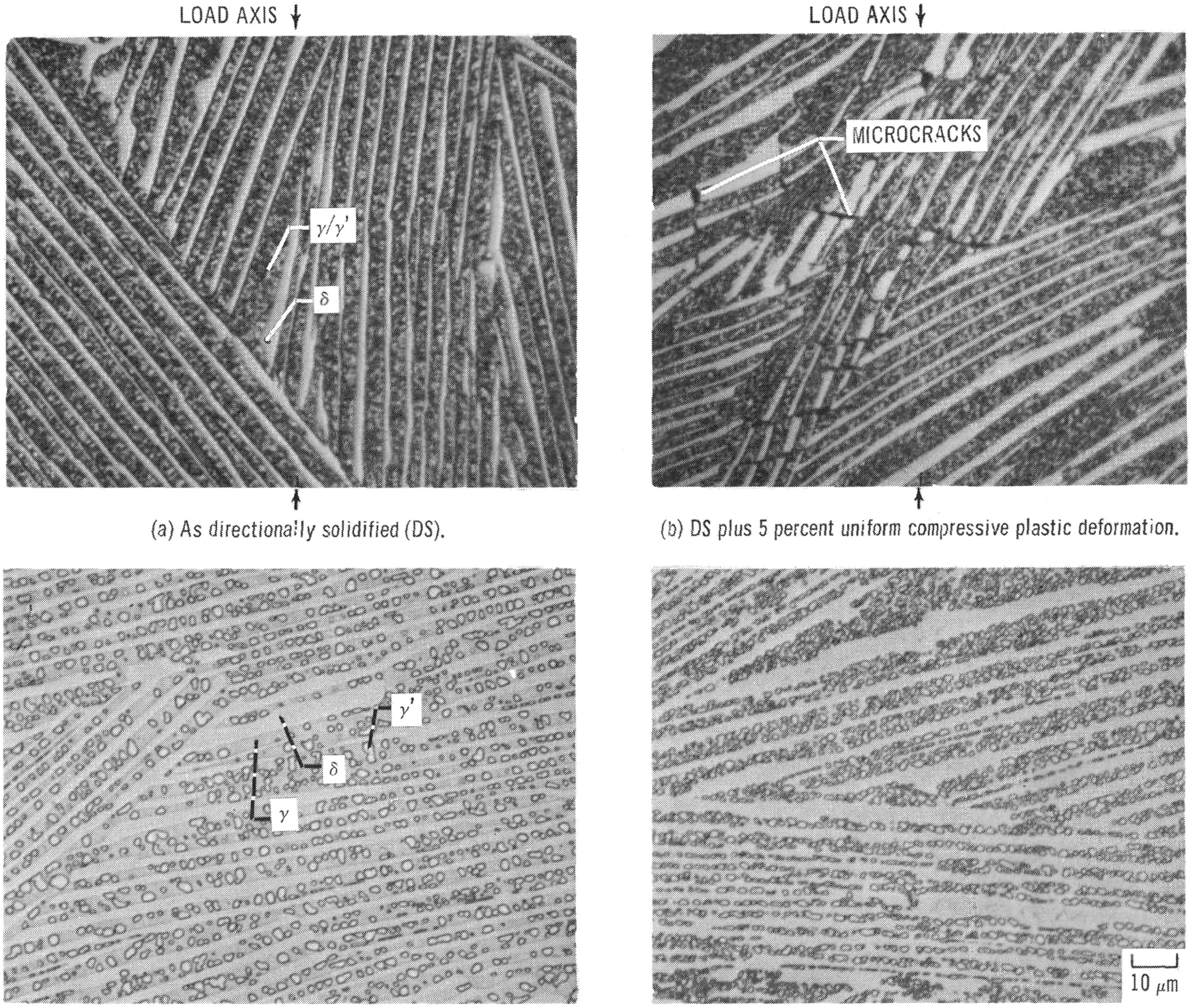


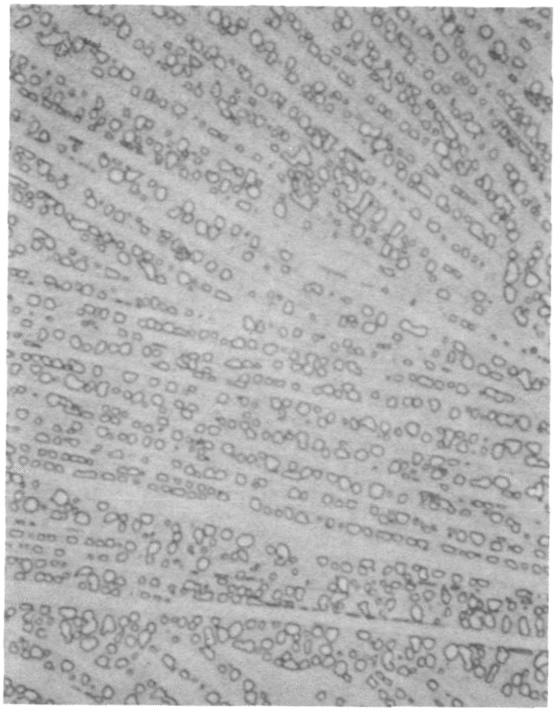
(a) As directionally solidified (DS).

(b) DS plus 5 percent uniform compressive plastic deformation.

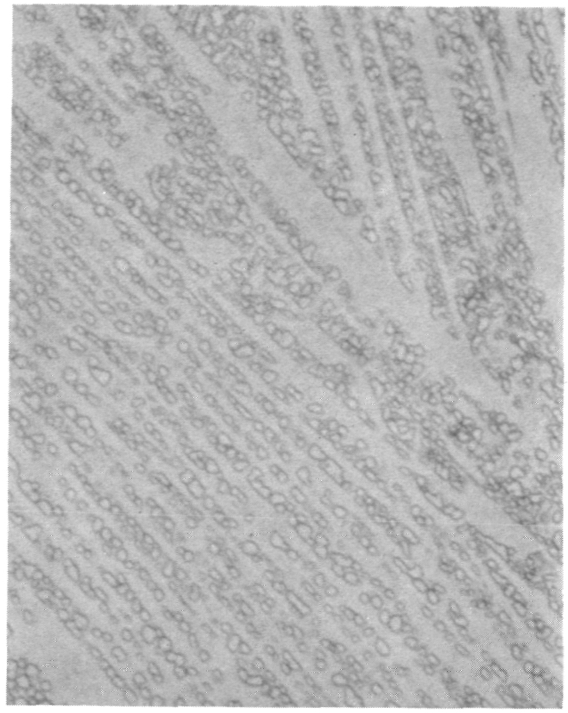
(c) DS plus 100-hour, 1100° C anneal.

(d) DS plus 5 percent deformation and 100-hour, 1100° C anneal.

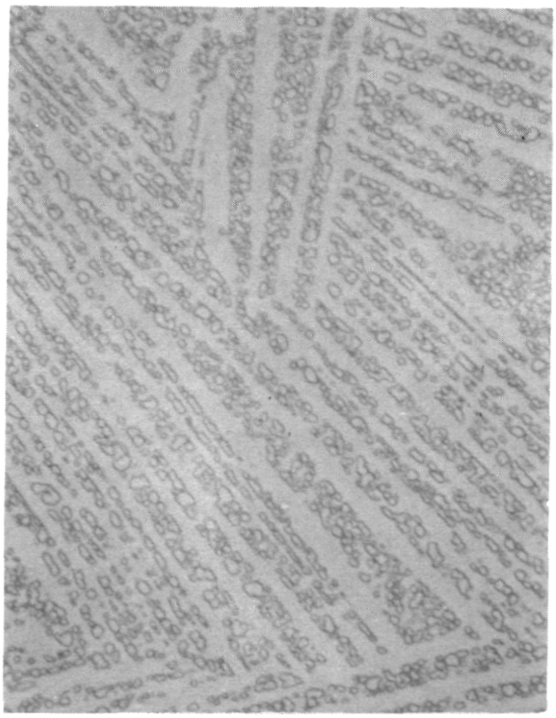
Figure 1. - Effect of 5 percent plastic deformation (load axis normal to the alloy growth direction) and subsequent 100-hour anneal at 1100° C on γ/γ'-δ eutectic alloy (transverse sections).



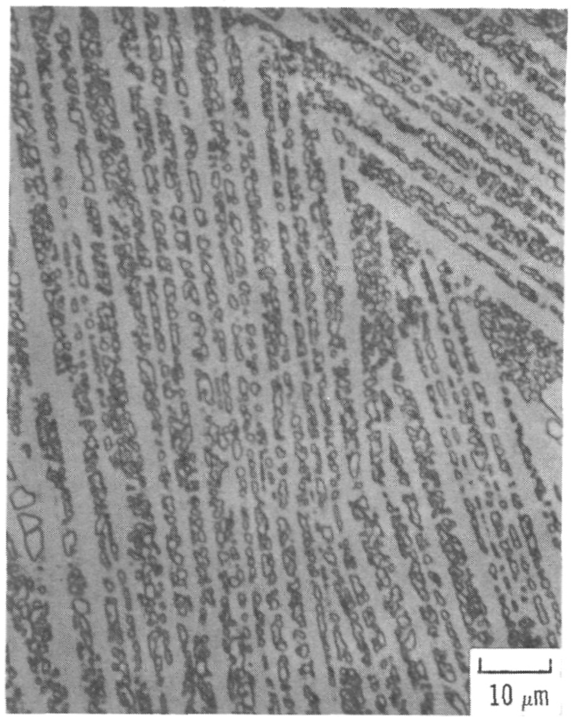
(a) As directionally solidified and annealed.



(b) DS + 1 percent plastic deformation + annealed.

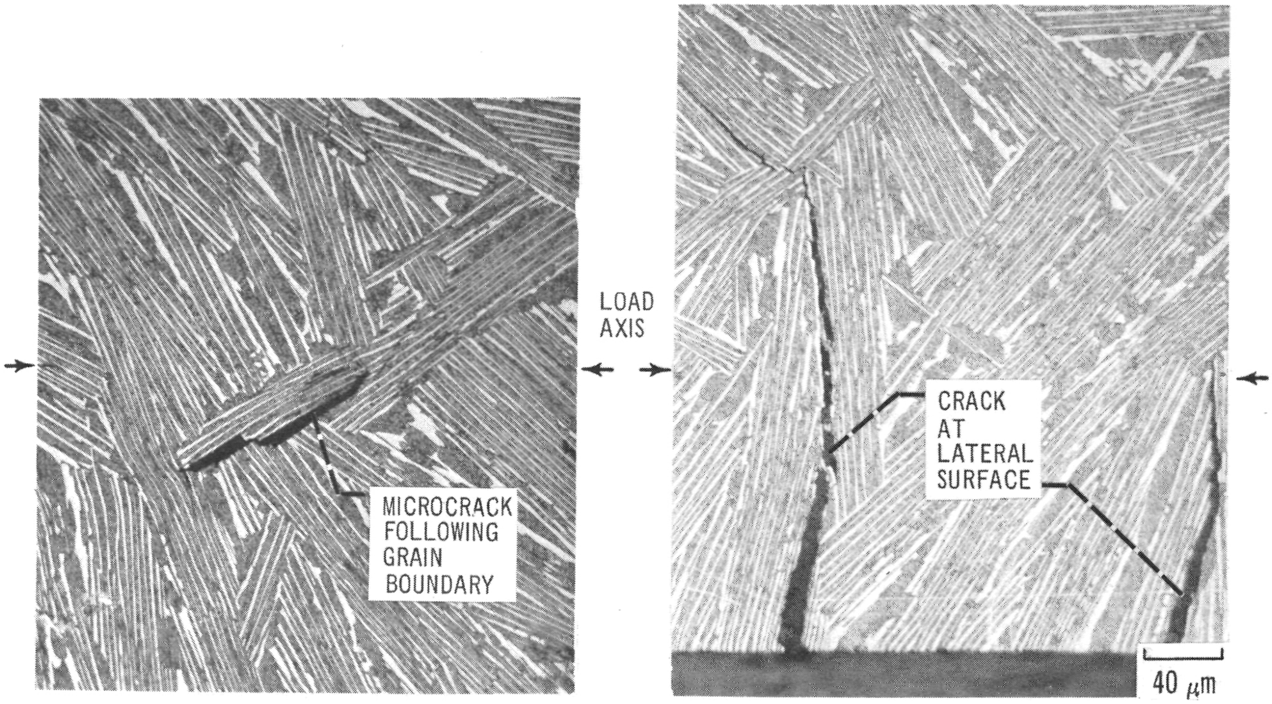


(c) DS + 3 percent plastic deformation + annealed.



(d) DS + 5 percent plastic deformation + annealed.

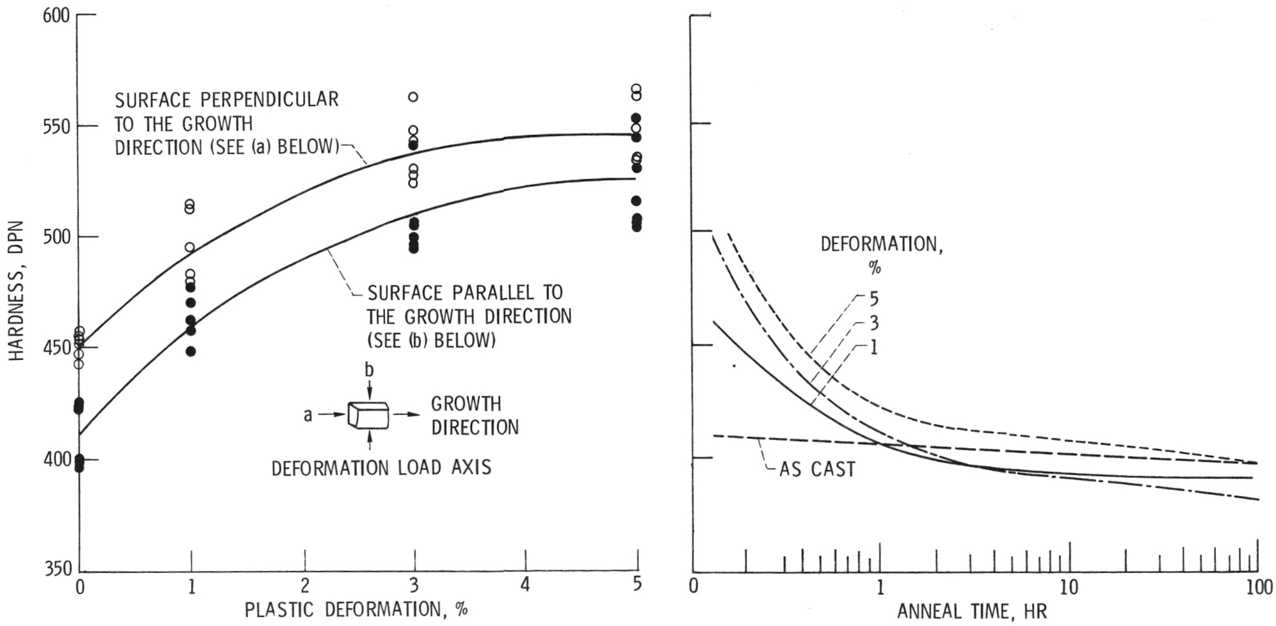
Figure 2. - Effect of uniform plastic deformation (load axis parallel to growth direction) on γ' growth in directionally solidified γ/γ'-δ eutectic alloy due to 100-hour, 1100° C anneal (transverse sections.)



(a) Internal crack.

(b) Surface crack.

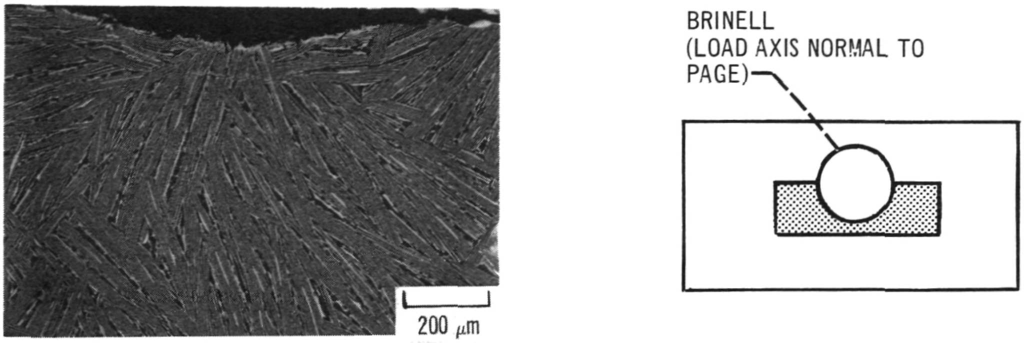
Figure 3. - Mode of cracking due to 5 percent uniform compressive plastic deformation (load axis normal to alloy growth direction) of γ/γ'-δ eutectic at room temperature (transverse sections).



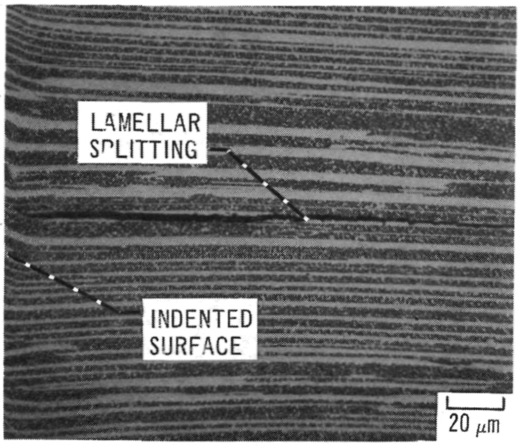
(a) Effect of uniform plastic deformation (load axis perpendicular to growth direction) on microhardness of two orthogonal surfaces.

(b) Change in microhardness as function of time (load axis perpendicular to growth direction; surface parallel to growth direction).

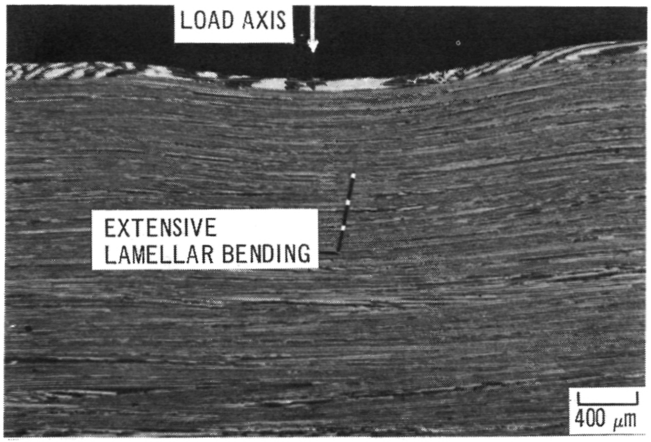
Figure 4. - Effect of uniform plastic deformation and subsequent 1100° C anneal on microhardness of γ/γ' - δ eutectic alloy. Diamond pyramid indenter load, 500 grams.



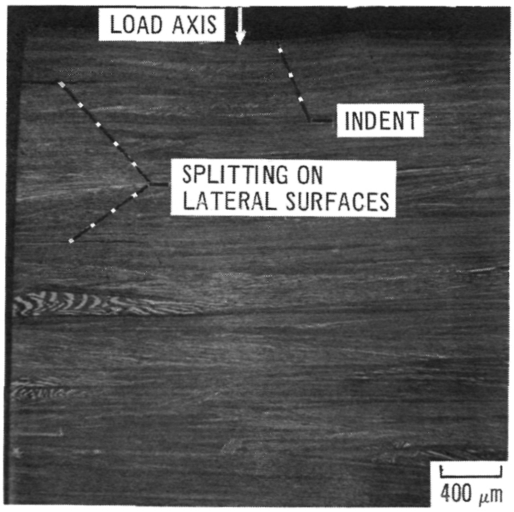
(a) Indented surface; slightly polished and etched. Load axis parallel to growth direction.



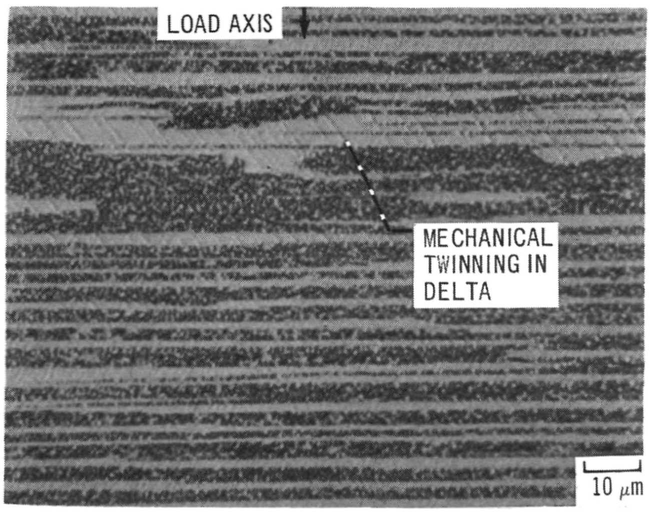
(b) Longitudinal section through indentation. Load axis parallel to growth direction.



(c) Longitudinal section through indentation showing severe lamellar bending. Load axis normal to growth direction.

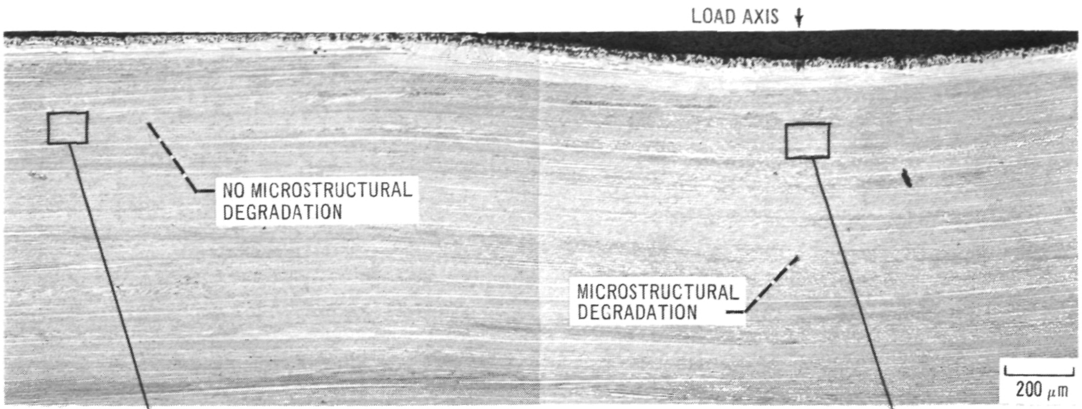


(d) Longitudinal section through indentation showing interlamellar splitting on surface parallel to load axis. Load axis normal to growth direction.

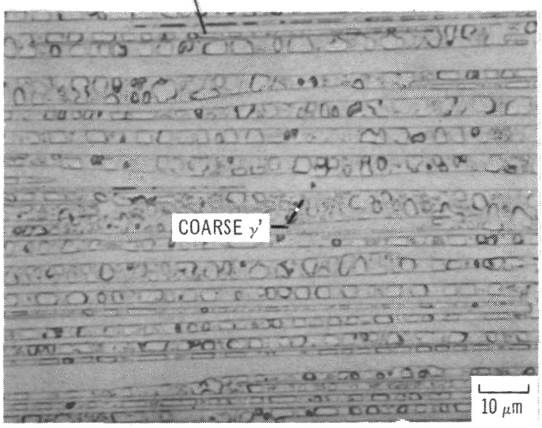


(e) Longitudinal section through region immediately below indentation. Load axis normal to growth direction.

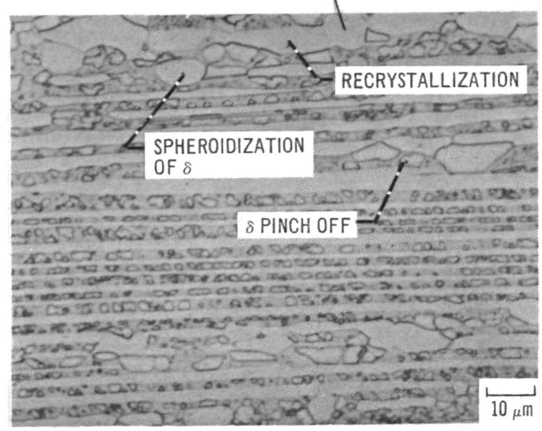
Figure 5. - Effect of localized plastic deformation due to Brinell indentation on microstructure of γ/γ' - δ alloy.



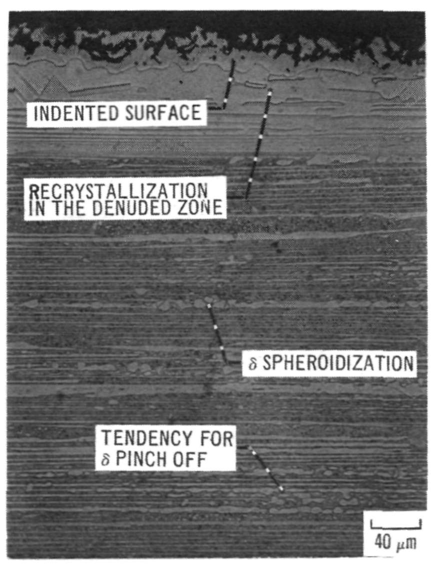
(a) Section through indented region.



(b) Absence of microstructural degradation away from severely deformed region.



(c) Microstructural degradation in severely deformed region directly below indentation.



(d) Section through severely deformed region directly below indentation.

Figure 6. - Degradation of the γ/γ'-δ microstructure due to Brinell indentation (load axis normal to the growth direction), followed by 300-hour 1120° C anneal (longitudunal sections).

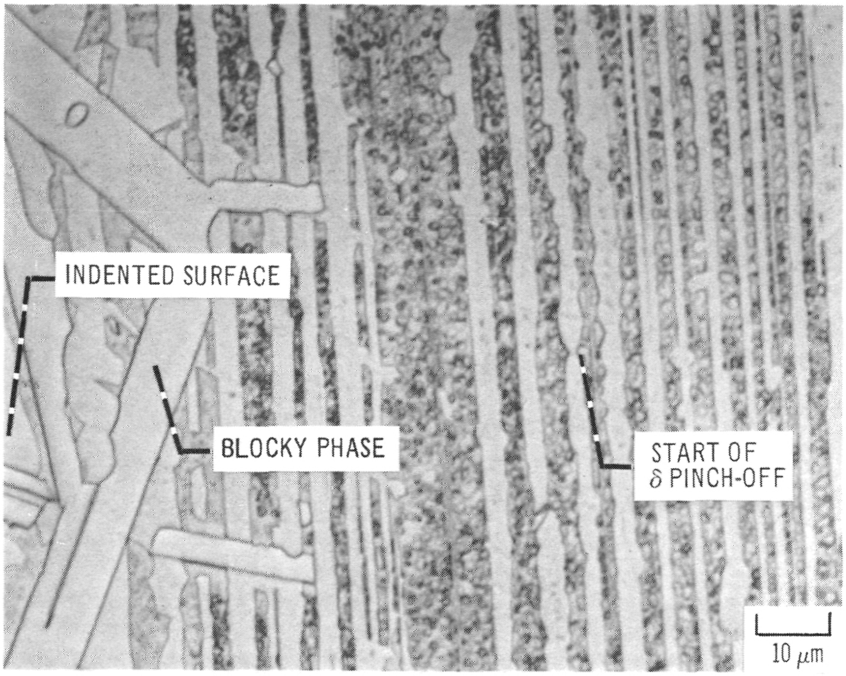


Figure 7. - Longitudinal section through Brinell indentation (load axis normal to growth direction) after 300-hour 1040° C anneal. Note formation of blocky phase and tendency for pinch-off of δ lamellae just below indented surface.

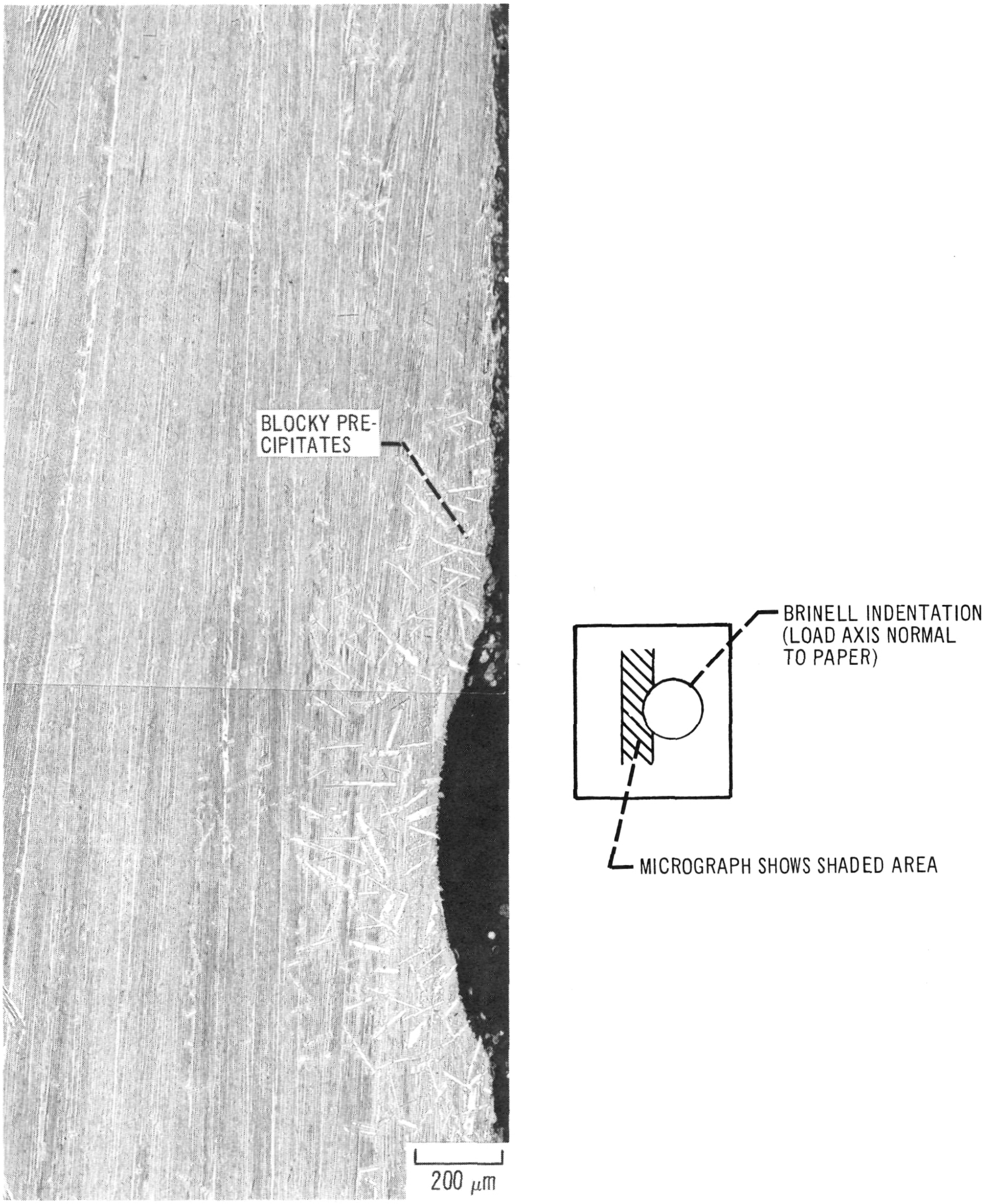


Figure 8. - Precipitation of blocky phase in plastically deformed γ/γ'-δ eutectic alloy surface after annealing at 1040° C. Indented surface after light polish and etch. Load axis perpendicular to growth direction; 300-hour, 1040° C anneal.

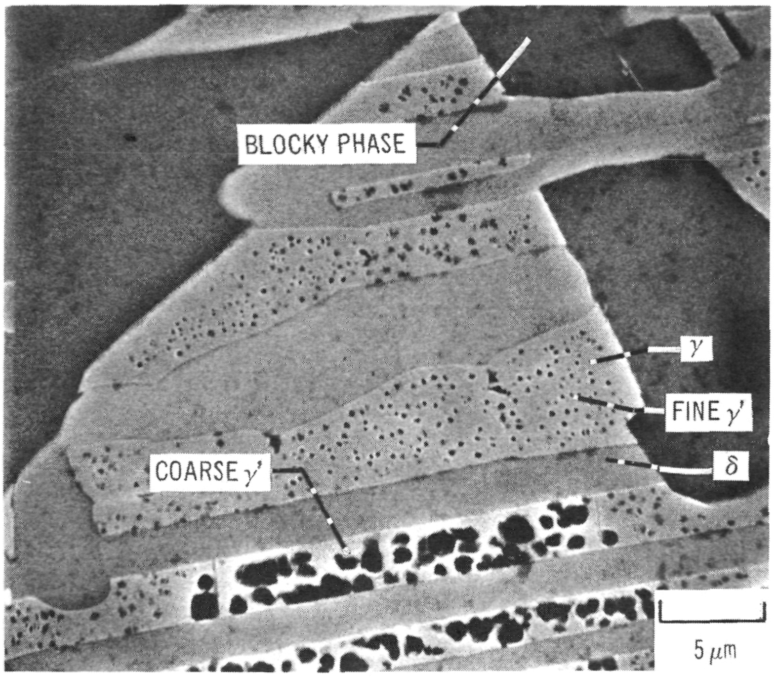
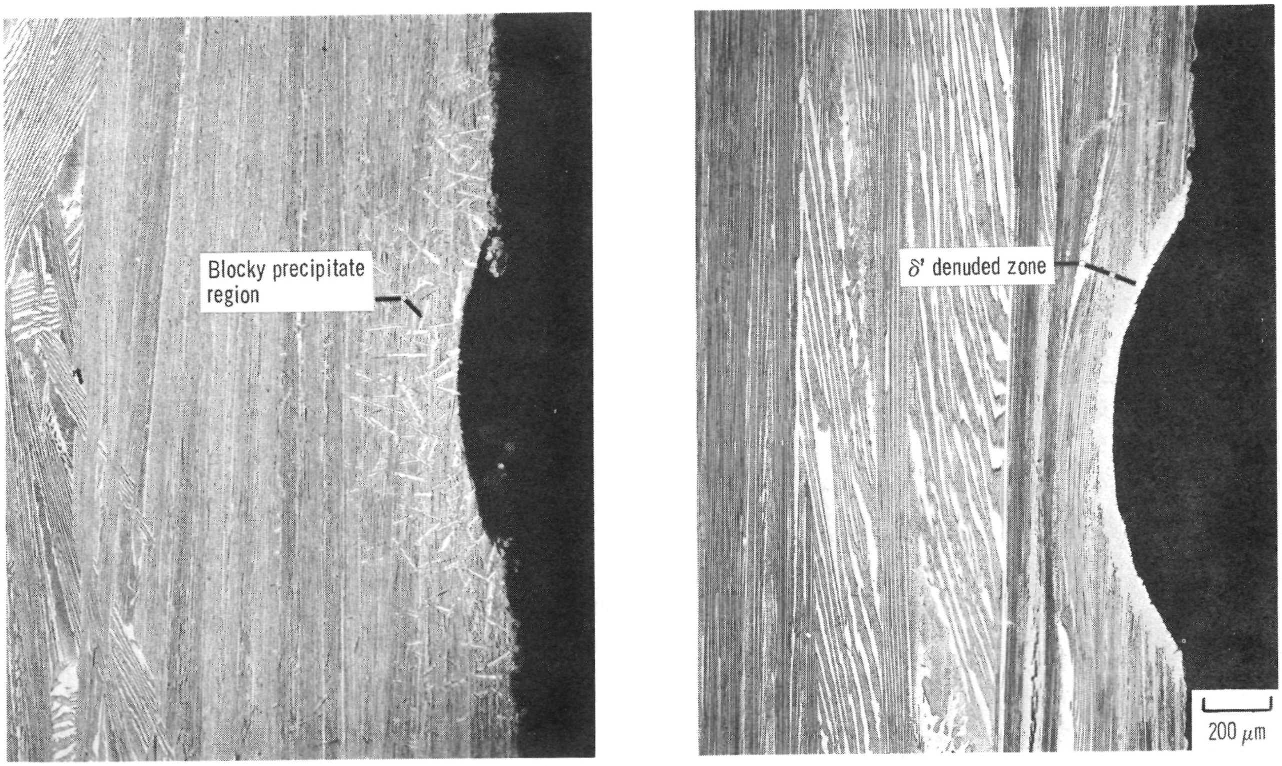


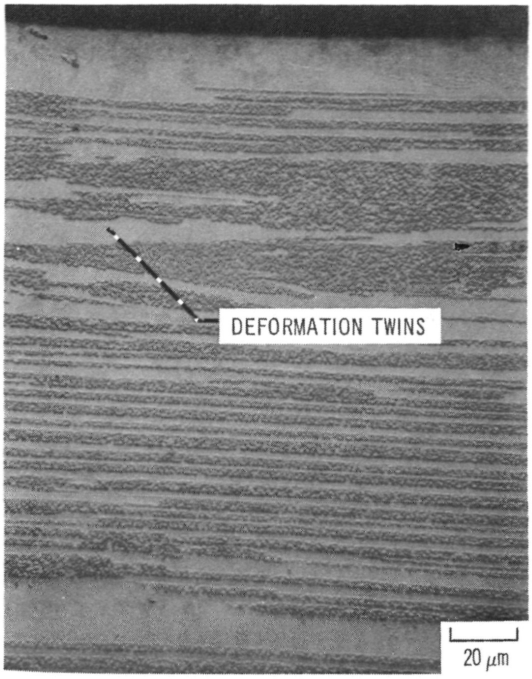
Figure 9. - Scanning electron micrograph (secondary-electron mode) of blocky phase on indented surface after polish and etch. Note fineness of γ' close to blocky phase areas. Load axis normal to growth direction; annealed 300 hours at 1040° C.

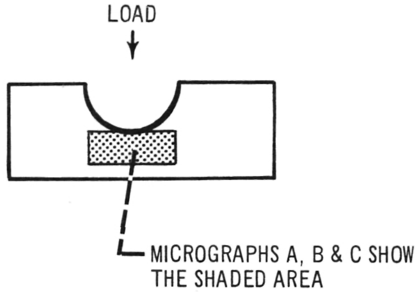


(a) 300-Hour 1040° C anneal.

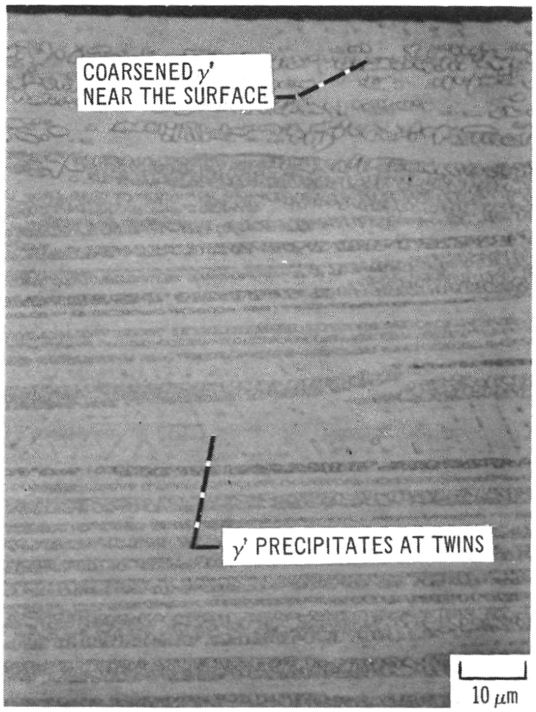
(b) 100-Hour 1120° C anneal.

Figure 10. - View of indented surfaces, slightly polished and etched to reveal structure after high-temperature anneal in argon. Load axis perpendicular to growth direction.

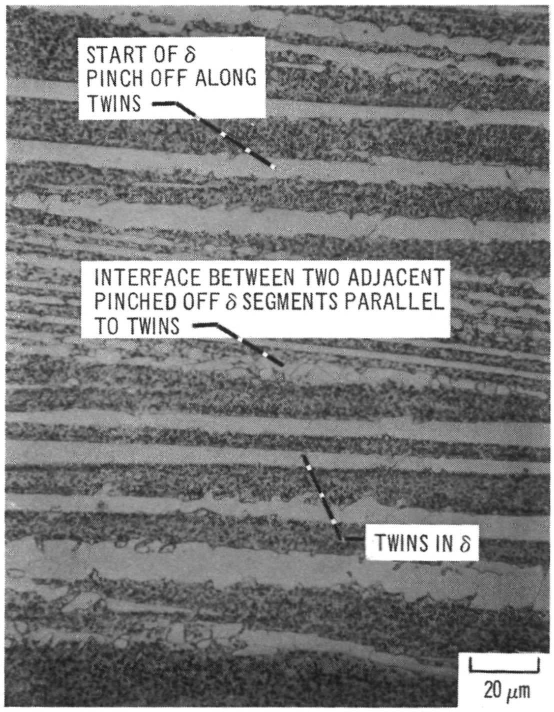




(a) 30 hours, 705° C.



(b) 30 hours, 1040° C.



(c) 30 hours, 1120° C.

Figure 11. - Microstructural change in γ/γ'-δ due to the Brinell indentation (load axis perpendicular to the growth direction) followed by 30-hour anneals at various temperatures.

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