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《Original》

A Study of the Slip Structure of an Aged and Deformed Nickel-Base Superalloy

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Abstract

Deformation modes at room temperature and the interaction between slip lines and γ' precipitate particles in heat treated René 41 were studied by means of replica techniques in an electron microscope. The alloy had been deformed by compression after aging for times up to 9300 hr at 760°C and 870°C.

The slip structure observed was similar to that of α brass, i.e., consisting of single slip lines occurred on the {111}<110> system. The slip structure was also correlated with aging conditions. Slip lines appeared to pass through γ' particles, shearing them with the matrix even after a prolonged heat treatment. It was concluded on the basis of comparison with previously published data that γ' particles remain at least partially coherent with the matrix even in a greatly overaged condition.

요 약

닉켈基 耐熱合金의 一種인 René 41을 760°C 및 870°C에서 最高 9300 時間까지 時効熱處理한 다음, 5% 내지 20%로 壓縮變形시켜, 이 合金의 室温에서의 塑性變形方式 및 析出粒子인 γ' 粒子와 slip line 間의 相互作用을 電子顯微鏡的 方法으로 觀察硏究하였다.

本合金의 slip 構造는 α 黃銅 때와 類似하게 {111}<110>系에 屬하는 單一 slip line으로 構成되어 있었으며, 純알루미늄에서 觀察되는 層狀構造는 볼 수 없었다. 또한, slip 構造는 時効條件에 따라 變化한다는 것을 알 수 있었다.

未時効狀態뿐만 아니라 過時効狀態에 있어서도 slip line은 如前히 γ' 析出粒子를 通過하였으며, 이 때 析出粒子는 地金과 더불어 變形되는 것을 볼 수 있었다. 이 觀察結果를 앞서 發表된 資料와 比較檢討하여 過時効段階에서도 γ' 粒子는 적어도 一部 地金과 整合되어 있다는 結論을 얻었다.

Introduction

It is well known that high temperature properties of most of nickel-base superalloys are influenced by the dispersion of precipitated particles of an ordered f.c.c. phase γ' (basically, Ni_3Al) in the f.c.c. solid solution matrix. The relationship between the dispersion of precipitated particles and mechanical properties of these alloys, however, has not completely established. In particular, since coherency strains play a major role in determining mechanical properties of certain nickel alloys[1], it is of a great importance to determine the coherency state of the γ' particles at various stages of aging process.

Thomas and Nutting[2] studied the interaction of slip lines with precipitated particles in aluminium-copper and aluminium-magnesium alloys. They observed that slip lines pass through coherent particles and partially coherent particles, shearing them with the matrix. They observed, however, that slip lines bend around non-coherent particles by a cross-slip mechanism without causing them to be deformed.

Madden et al[3] used a similar technique for a study of deformation of a nickel-base superalloy and observed that slip lines appeared to pass through γ' particles even in overaged specimens. They did not conclude, however, that precipitated particles are coherent with the matrix but suggested that slip lines did not bend around the particles because the necessary cross-slip mechanism was not thermally activated at room temperature.

In recent years, several workers[4,5,6] investigated the deformation of nickel-base superalloys by transmission electron microscopy. It showed that coherent particles were cut by dislocations and sheared with the matrix on deformation. These studies, however, placed their emphasis on the particle-dislocation interaction in underaged or slightly overaged specimens, and there are only a few available papers on the study of interaction in the fully overaged condition.

The present study was undertaken in an attempt to observe the deformation behavior of heat treated René 41 at various stages of aging process and at various strain levels, and to determine the state of coherency of γ' precipitated particles, particularly in the fully overaged condition, by observation of the interaction between slip lines and γ' particles.

Experimental Procedures

René 41 was selected for this investigation. Rectangular blocks of approximately 12 x 10 x 20 mm in size were cut from a forged bar stock of a commercial heat having the following weight composition: 0.09% C, 0.05% Mn, 0.10% Si, 18.60% Cr, 10.80% Co, 10.00% Mo, 3.22% Ti, 1.80% Al, 1.40% Fe, 0.006% B, 0.015% P, 0.006% S and balance Ni.

They were given a solution treatment at 1150°C for 4 hr; followed by water quenching and then isothermal aging at 760°C and 870°C respectively, for times up to 9300 hr; followed by air cooling. Disks were cut from the heat treated blocks, and they were used for both hardness measurements and metallorgraphic examinations. Hardness values reported here represent the average of three or four readings. For the metallographic examination, specimens aged for less than 1000 hr were mechanically polished and etched by immersion in a solution of 92 parts by volume of hydrochloric acid, 5 parts of sulfuric acid and 3 parts of nitric acid. Specimens aged for more than 1000 hr were etched anodically in an aqueous solution of 10% oxalic acid after mechanical polishing to reveal grain boundaries of the specimens.

Heat treated rectangular blocks were electropolished in an electrolyte consisting of 10 parts of perchloric acid and 90 parts of glacial acetic acid after mechanical polishing and then deformed by compression from 5 up to 20% at the rate of 0.005 cm/min. at room temperature.

For the observation of the interaction between slip lines and γ' particles, specimens are lightly etched anodically in a solution consisting of 47 parts of sulfuric acid, 12 parts of phosphoric acid and 41 parts of nitric acid after electro-polishing. Double stage replicas shadowed with platinum and carbon were prepared for the examination in an electron microscope (Hitach HU-125C). Carbon replicas were also prepared to observe the surface structure in detail of plastically deformed specimens. A solution of 10% hydrochloric acid in ethyl alcohol was used to free the replicas from the surfaces of the specimens. Diameters of over 300 particles in the same specimen were measured to determine the size of γ' particles in the overaged condition.

Results and Discussion

1. Changes in Hardness and the Growth of γ' Particle at Different Aging Conditions

The effect of varying aging temperature and time on the hardness is shown in Fig. 1. The maximum value of the hardness was obtained after aging for 32 hr at 870°C and for 64 hr at 760°C, respectively. As expected, with raising the aging temperature, the peak hardness decreased, occurring at much shorter aging times. It is noteworthy that the hardness decreased very slowly from the maximum value even after a prolonged aging heat treatment, e.g., after aging for 9300 hr at 870°C.

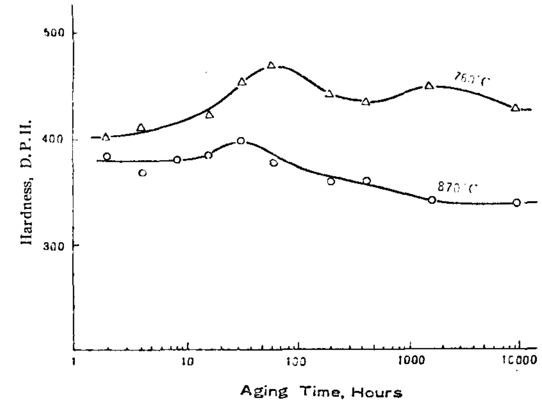


Fig. 1. Change in hardness of René 41 during aging at 760°C and 870°C.

As aging proceeded, numerous spheroidal γ' particles were precipitated within matrix grains, and no change in the shape of γ' particles was observed during the whole aging process. After aging was carried beyond the peak hardness, "needles" phase (Ni_3Ti) were observed, together with γ' particles growing coarser with further aging. The needles appeared to be growing in the {111} planes of the matrix since slip lines whose slip plane is the {111} planes, running parallel to these needles, were easily observed. The size and interparticle spacings increased with increasing time and temperature of aging. The variation in the particle size of γ' precipitates as a function of aging time are plotted in Fig. 2. Under a certain

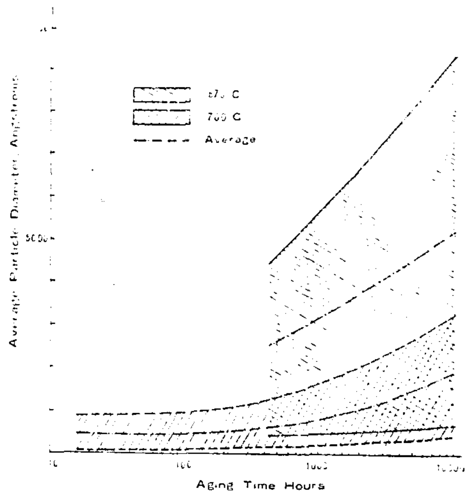


Fig. 2. Estimated range of size of γ' particles in René 41 aged at 760°C and 870°C.

aging condition, the distribution of γ' particles shows the normal distribution curve, and it is noteworthy that some parts of particles are retained from coarsening even after a long period of aging.

A log-log plot of particle size vs. aging time are shown in Fig. 3. Average diameters in-

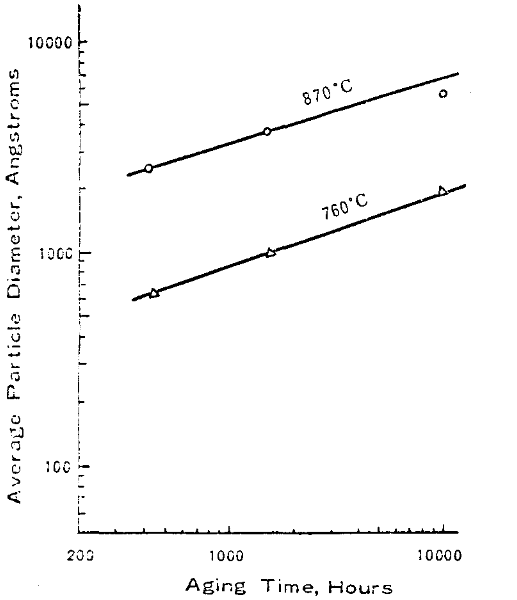


Fig. 3. Change in average particle diameter with aging time at 760°C and 870°C.

creased in proportion to the cube-root of the aging time beyond 400 hr. This result is in good agreement with that of Silcock and Williams[7]. The discrepancy at 870°C line are likely due to the massive formation of η phase that resulted in a random distribution of γ' particles.

2. Determination of Slip System

In order to determine the orientation of slip lines, a known orientation relationship that annealing twin boundaries lie on the {111} planes was used. In all aged specimens, abundant annealing twins were easily observed, and the orientation of slip lines would be easily determined if two differently oriented twins exist in a grain crystal.

Photo 1 (a) shows the slip traces in the vicinity of hardness impressions on polished surfaces, and Fig. 4 shows a stereographic projection corresponding to Photo 1 (a). The surface of the specimen was taken as the plane of projection. Surface traces of two twin boundaries, i.e., {111} planes in the matrix, T_1T_1, T_2T_2 and that of the activated slip plane, SS are shown around the circumference of the projection. In Fig. 4, P indicates the pole of the

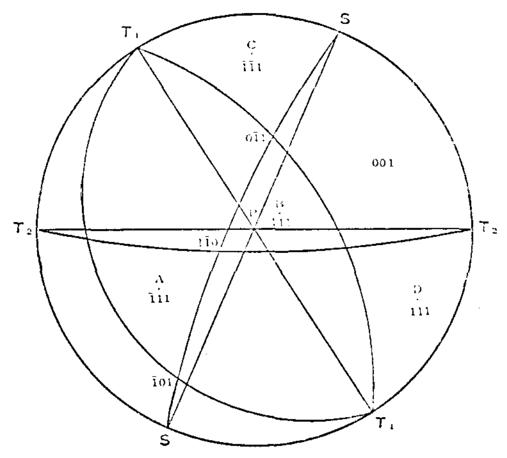


Fig. 4. Stereographic projection of the area shown in Photo 1 (a).

polished surface, and A, B, C are the poles of twin planes, and D is that of the activated slip plane, respectively. From the figure, it was easily found that the active slip planes are the {111} planes, and the slip directions are ⟨110⟩. There are three possible slip directions, i.e., $[\bar{1}01]$, $[1\bar{1}0]$, $[0\bar{1}1]$ on the slip plane. In order to determine the exact slip direction, the change of shape of the precipitate which has a definite crystallographic orientation should be known. Unfortunately, this condition was not fulfilled in the case.

3. Slip Characteristics of the Alloy

To observe the influence of aging heat treatment upon slip characteristics of the alloy, optical observations were made on slip traces adjacent to Rockwell "C" hardness penetrations in polished surfaces.

In the as-quenched and short-time aged state, slip lines were composed of sharp, single steps of large displacement, and they mostly extended from grain or twin boundaries to grain or twin boundaries. With approaching the peak hardness, slip lines tended to become more finer and less distinct with varying depths and tended to be distributed at random. Examples are shown in Photos 1 (a) and (b).

Even in the same specimen, the number of activated slip systems varied according to the orientation of the crystal so as to accommodate the complicated plastic deformation of the polycrystals. The number of activated slip systems at a certain strain decreased with increasing hardness, i.e., four slip systems activated in the short-time aged specimens, whilst only two slip systems activated in the specimens around the peak hardness.

Comparing slip displacement occurred in the grains adjacent to the impression with those in grains apart from the impression, no constant relationship between the slip displacement and the amount of strain was found. Same results were obtained from an electron microscopic study.

For the purpose of studying the slip structure in details, electron microscopic observations were made on specimens compressed up to 20% by increments of 5% at room temperature.

In general, slip occurred on individual slip planes that were relatively widely spaced with varying depth for small strains, and no regular lamellated formation of slip bands as observed in high purity aluminium were found. Occasionally, groups of more or less uniform depth were found that is similar to slip bands, as shown in Photo 2. However, these quasi-lamellar type slip lines were very rare and only exist in a narrow region and were far from regularity as found in aluminium.

With increasing deformation, the number of quasi-lamellae increased from two to five when strain increased from 5 to 20%, and no distinct influence of heat treatment on the number of those were found.

It was extremely difficult to correlate the spacings of coarse slip lines with heat treatment conditions because the spacing of slip lines on each specimen varied very greatly and even the regions as wide as 9 microns on which not a trace of slip could be detected for 5% strain. Thus, it seemed reasonable that the spacings of slip lines are not related with heat treatment, and it is apparent from the observation that the wide regions, free of slip lines, are gradually filled with slip lines or quasi-lamellae with increasing deformation.

In contrast to this, evidences showed that slip displacement is closely related to the aging condition, i.e., slip displacement decreases with approaching the peak hardness. Unfortunately, the amount of slip was not shown here quantitatively, since the orientation of individual crystals was not known.

Usually, slip lines appeared to be homogeneous especially in the short-time aged condition. However, a close examination revealed that abruptly discontinuous slips occurred very often as shown in Photo 3. This may be inferred that slip lines had altered its slip plane by the mechanism of cross-slip or alternatively, a neighboring slip system had begun to act by the piled-up dislocations in the slip plane activated earlier.

In the surfaces of samples deformed by compression, it was observed less frequently that slip lines appearing light were intermixed in a set of parallel lines that were dark as shown in Photo 4. Such color differences indicate that slip had not occurred in a normal manner as illustrated schematically in Fig. 5 (a) but in a manner as shown in Fig. 5 (b).

The fact that there are wide regions, where no slip traces are detected, may be interpreted that the regions between slip lines contain no or very few dislocations, and most dislocations would be arranged in a coplanar plane at room

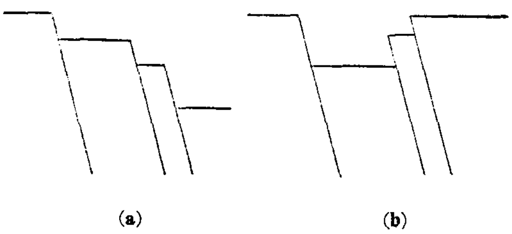


Fig. 5. Diagram illustrating the formation of slip lines by compression, (a) normal mode, (b) peculiar mode (refer to Photo 4).

temperature deformation, since it was not difficult to reproduce strain markings on the nickel-base superalloy by etching the repolished surface of a deformed specimen so as to reveal slip trace. Gell and Field [8] have shown that an etching technique can be applied for revealing slip traces of deformed specimens in which interstitial diffusion was not involved. In recent years, Beardmore et al [6] have observed, in the study of nickel alloys, narrow bands of densely packed dislocations by direct observation at room temperature deformation.

Multiple slip was observed in all specimens deformed beyond 5%. By geometrical orientation of the crystals to the direction of applied stress, the most highly stressed system begins to act alone. As deformation proceeds, critical resolved shear stress becomes high enough to activate the second system consecutively, but not simultaneously. Photo 5, in which Burgers vector is supposed to be parallel to the surface, shows clearly that slip lines formed earlier were sheared or branched off by following slip lines formed later, and thus the figures denoted on the micrograph indicate the order as slip systems had activated successively. Of course, this does not rule out the possibilities that multiple slip can occur simultaneously due to the geometrical symmetry of individual crystal orientations. Generally, slip lines formed earlier show more deeper and distinct than those of slip lines formed later, and this may be attributed in part to the influence of slip lines formed later on those formed earlier.

In some slip line patterns, mostly in the case of multiple slip, wavy slip lines were observed, for instance, as shown in Photo 6. The reason may be that slip lines formed earlier were cut very often by lines of crossing system, and the severely distorted surface on which they meet influenced the appearance of slip patterns. At any rate, no wavy slip lines as found in b.c.c. metals were observed.

Annealing twins were abundantly observed in all the specimens examined. As shown in Photo 7, slip lines approaching twin boundaries were either fading out or stopped at boundaries abruptly. Even in the case, as shown in Photo 8, where slip lines appeared to pass through boundaries directly with a change of direction, a close observation revealed frequent disturbances at twin boundaries. From these observations, it is evident that twin boundaries exhibit an obstacle for crossing slip lines. Wilsdorf et al [9] have already put forward this conclusion.

4. Cross-slip

In all the specimens examined, cross-slip was abundantly observed, though cross-slip occurred less frequently with increasing hardness. In a same specimen, cross-slip occurred either near grain boundaries as shown in Photo 9 or within the grain as shown in Photo 10 due to the random distribution of stresses in the polycrystals. Especially at high deformation, all lines of one system, as a whole, turn into the cross-slip system, and as a result it was difficult to distinguish which lines are cross-slip lines and which lines are not. The examples are shown in Photos 11 and 12.

A special type of cross-slip reportedly observed in α brass [9] was also observed in this alloy, as shown in Photo 13, which existed in the immediate neighborhood of etch pits. It shows that slip lines traversing the pit are clearly continuous and that cross-slip does not extend beyond the pit.

In a recent paper, Kotval et al[10,11] found that most nickel-base superalloys fall within the category of "medium" stacking fault energy class based on the type of dislocation substructure observed in the matrix. In this category the stacking fault energy is not sufficient enough to make the dissociation of dislocations. Thus, it results in coplanar groupings of dislocations. However, with successive addition of solute in the matrix, there is an increased tendency toward dissociation of dislocations, and cross-slip becomes progressively more difficult to occur. According to the above observation, the alloy René 41 may be placed under the category of "medium" or at least under the transition category from "medium" to "low" stacking fault energy. The conclusion made in the preceding section that dislocations are likely arranged in the coplanar plane suggests strongly that the alloy may fall into the category of "medium" and that cross-slip is possible to occur. Even though the alloy is assumed to be a boundary case, the width of stacking fault band would be narrow enough to enable dislocations cross-slip. From this consideration, it may be concluded that cross-slip is possible to occur at room temperature deformation in this alloy.

5. The Interaction of Slip Lines with γ' Particles

In an underaged condition before reaching the peak hardness, e.g., after aging for 16 hr at 760° C, the size of γ' particles was of the order of 500 Å on the average. Owing to the lack of resolution in replicas, it was sometime difficult to judge whether slip lines pass through γ' particles or stop at them. An evidence that slip lines pass through the particles was obtained, however, as shown by an example given in Photo 14.

When aging was carried out beyond the peak hardness, e.g., after aging for 64 hr at 870°C, the particle size ranged up to a few thousand Å, and the interaction between slip lines and γ' particles was easily boserved. In general, slip lines appeared to pass through γ' particles even for small strain, say 5%, which is lower than the yield strain, but sometimes appeared to bend around the particles at diffierent locations of a same specimen. When a specimen was deformed at the yield stress, most of slip lines passed through γ' particles and slip lines bending around γ' particles, as shown in Photo 15, were rarely observed. When deformation increased, however, beyond the yield stress, many more slip lines passed through γ' particles, as shown in Photo 16, and some γ' particles sheared severely with the matrix, as shown in Photo 17.

Whether slip lines pass through γ' particles or bend around them for small strain, depends on the relative orientations of the particles, slip planes and directions. This is so because the distribution of stress within a polycrystalline specimen is inhomogeneous. Slip lines on a greatly stressed slip system may pass through γ' particles, whereas they may not on a weakly stressed slip system. A careful examination of Photo 18 shows that slip lines belonging to the slip system activated initially pass through γ' particles, while most of those fine slip lines belonging to the slip system activated later either stopped by γ' particles or bent around γ' particles. This apparently suggests that γ' particles show a resistance to deformation of a low stress level. In further support of this evidence, there was a general tendency for slip lines to stay away from γ' particles and to be arranged in between γ' particles. Furthermore, frequent occurrence of cross-slips around γ' particles was observed, as shown in Photo 19.

In order to study the interaction between slip lines and γ' particles in a greatly overaged condition, observations were made on slip traces in the region adjacent to a hardness impression after aging for 9300 hr at 870°C. In this condition, the maxium particle size increased to nearly 9000 Å. It was found that slip lines still cut through γ' particles, as shown in Photo 20, and extensive shearing of particles sometimes occurred, as shown in Photo 21. It was also observed by the examination of Photo 22 that fine slip lines were either stopped short of γ' particles or concentrated in the rather spacious region between γ' particles. From the above observations, it may be concluded that most of γ' particles in the overaged condition initially resist shearing for a small strain but are deformed eventually for a large strain.

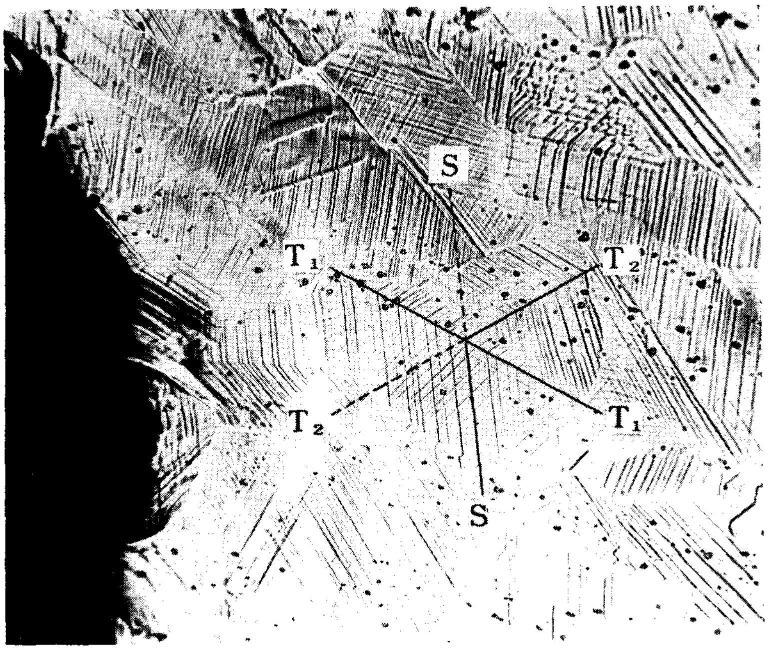
The conclusion of Thomas and Nutting[2] was based on observations of surfaces of heavily deformed aluminium alloys, say, the region adjacent to the fracture. Considering this and the observations in the present study that γ' particles are cut through by slip lines even for a small strain ranging 5 to 20%, it may be concluded that γ' particles remain at least partially coherent with the matrix over the entire range of aging examined. It is a known fact that[12] non-coherent particles of an oxide or intermetallic compound do not generally appear to deform at all if they are of size up to a few microns. Madden et al[3] obtained similar results from a study of nickel alloy aged up to 250 hr at 870°C and strained 12 to 15% by elongation. They alternatively suggested that γ' particles actually may not be coherent with the matrix but that slip lines did not bend around the particles because the necessary cross-slip mechanism was not thermally activated at room temperature. As discussed in a preceding section (Section 4.), cross-slip is possible to occur in this alloy at room temperature deformation as opposed to their suggestion. An evidence that clearly shows slip lines bending around the non-coherent particles at room temperature was actually obtained, as shown in Photo 23.

It is very interesting to note that the drop in the yield stress of samples in an overaged condition is far more greater than that of hardness. The rapid decrease in the yield stress may be interpreted as follows; in the early stage of aging, coherent precipitate particles are so closely spaced that dislocations tend to pass through the particles rather than to bow between them. As aging proceeds beyond the peak hardness, the interparticle spacing increases with coarsening of particles with accompanying partial loss of coherency at the interface between the particles and the matrix. As a consequence, it becomes much easier for dislocations to bow between them, resulting in a decrease in the yield stress. When dislocations bow between particles by the process of cross-slip, dislocation loops may be left around the particles. It was suggested that the number of loops increases with increasing deformation, and as the stress becomes sufficiently great, some loops may pass through the particles, causing shearing of the particles. In fact, some examples[7,13] that show dislocation loops encircling precipitated particles in austenitic steel in an overaged condition have been recently observed by transmission electron microscopy. Thus, it may be concluded that the concept developed by Fisher, Hart and Pry[14] is applicable to the interaction of slip lines with particles in overaged and deformed nickel-base superalloy, René 41.

Summary and Conclusions

The experimental results led to the following conclusions:

1) In an overaged condition, the size of γ'



(a)

(b)

Photo 1. Slip traces adjacent to Rockwell "C" hardness penetrations in polished surface of heat treated René 41; (a) Aged for 2 hr at 870°C, ×200, (b) Aged for 64 hr at 870°C, ×200.

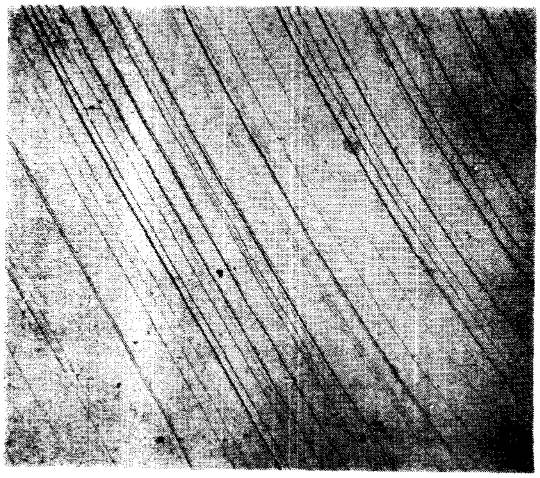


Photo 2. As quenched, reduced 20%, ×1,000.

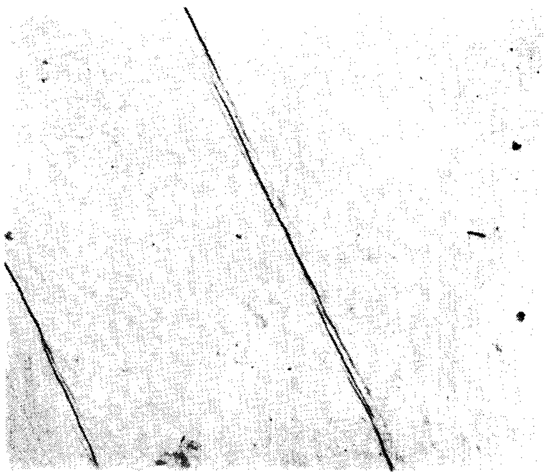


Photo 3. As quenched, reduced 10%, ×10,300.

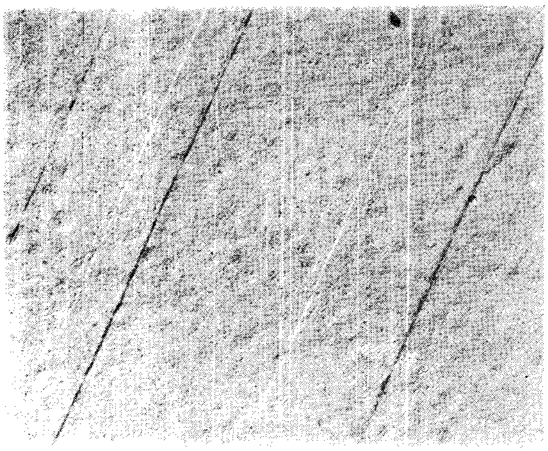


Photo 4. As quenched, reduced 15%, ×9,000.

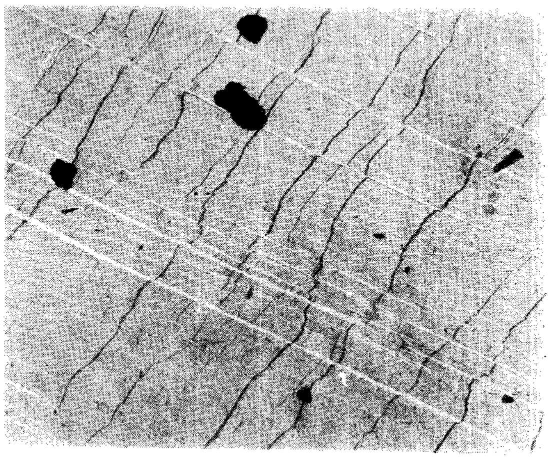


Photo 6. As quenched, reduced 20%, ×7,000.

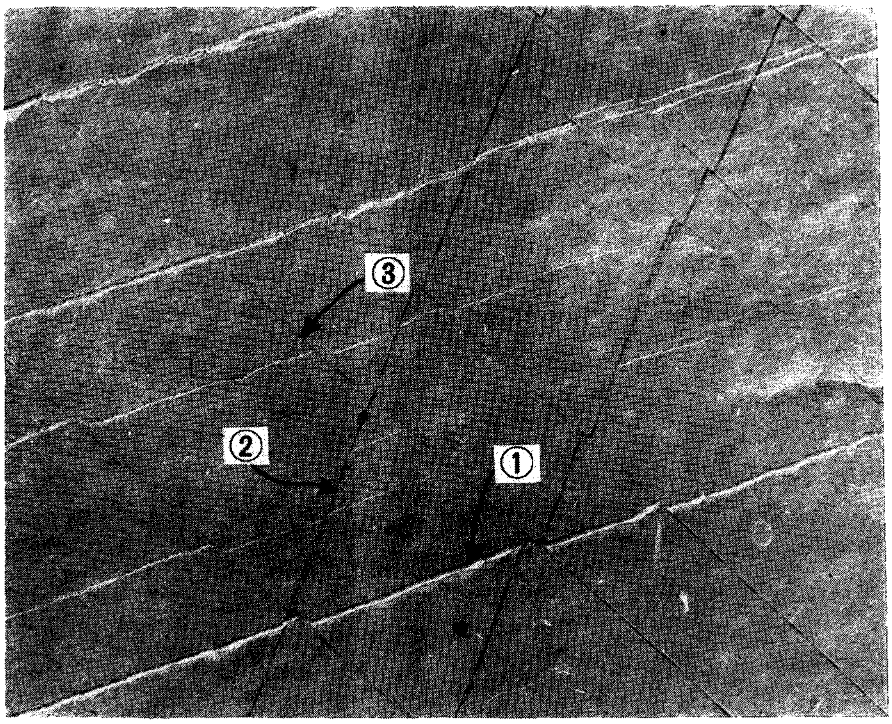


Photo 5. As quenched, reduced 15%, ×15,000.

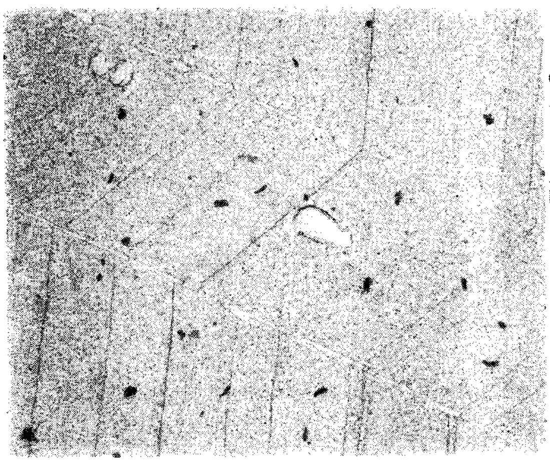


Photo 7. Aged for 5 hr at 760°C, reduced 20%, ×2,500.

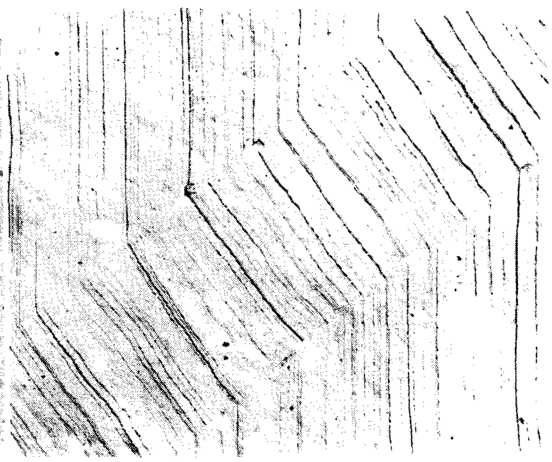


Photo 8. Aged for 32 hr at 870°C, reduced 20%, ×2,000.

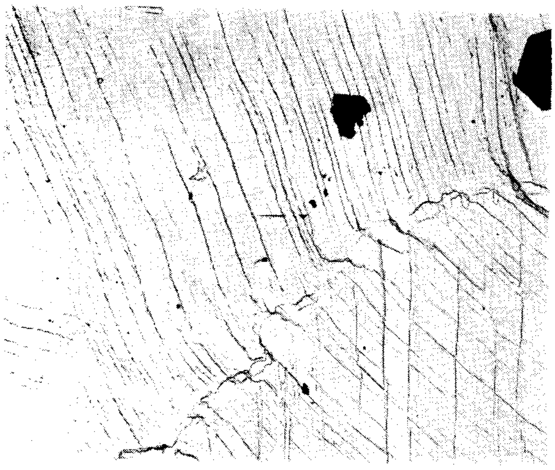


Photo 9. Aged for 64 hr at 870°C, reduced 20%, ×3,500.

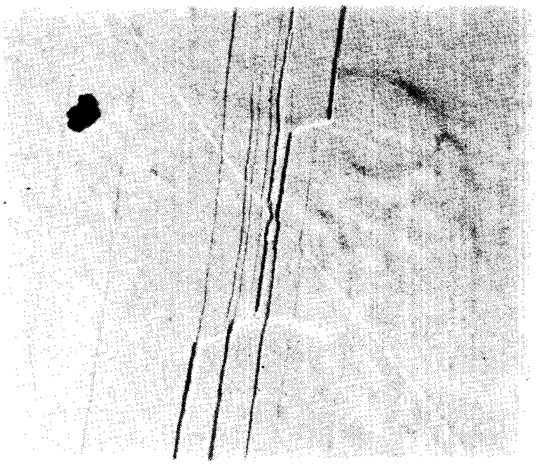


Photo 10. As quenched, reduced 10%, ×10,000.

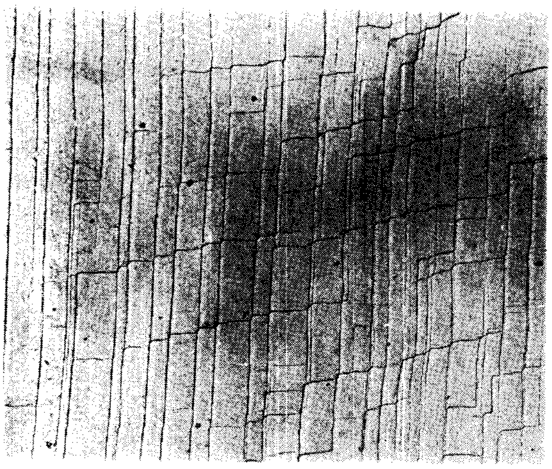


Photo 11. Aged for 32 hr at 870°C, reduced 20%, ×4,000.

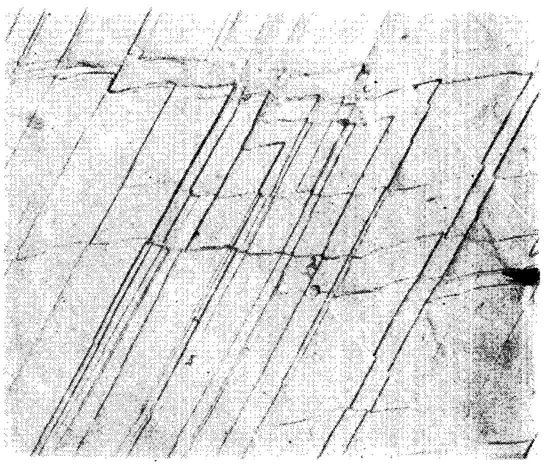


Photo 12. As quenched, reduced 20%, ×9,500.

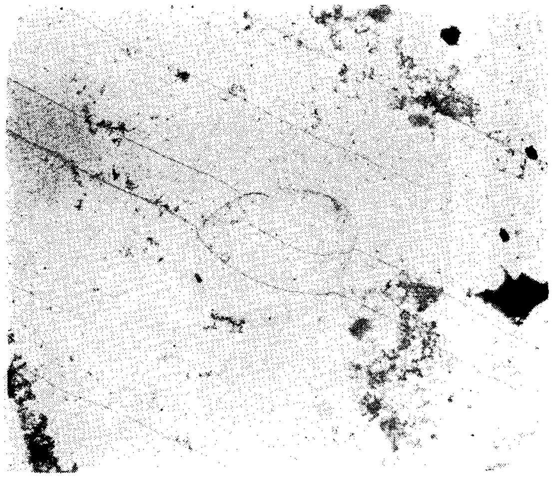


Photo 13. Aged for 8 hr at 760°C, reduced 20%, ×5,000.

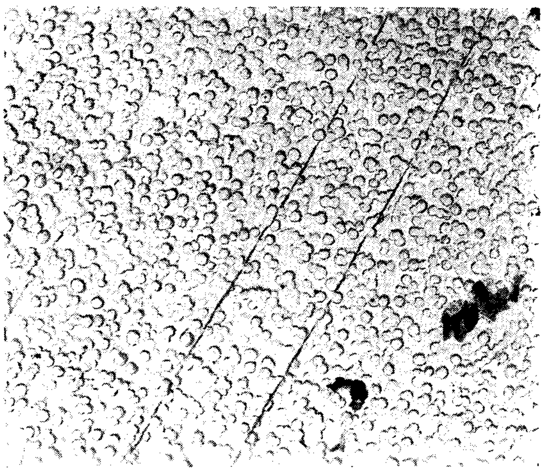


Photo 14. Aged for 0.5 hr at 760°C, reduced 20%, ×25,000.

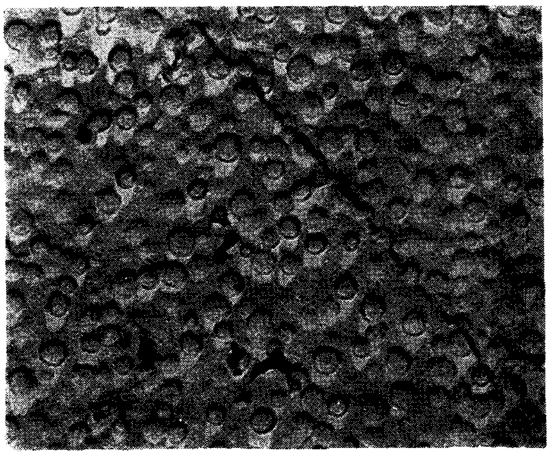


Photo 15. Aged for 215 hr at 760°C, reduced at the yield stress, ×30,000.

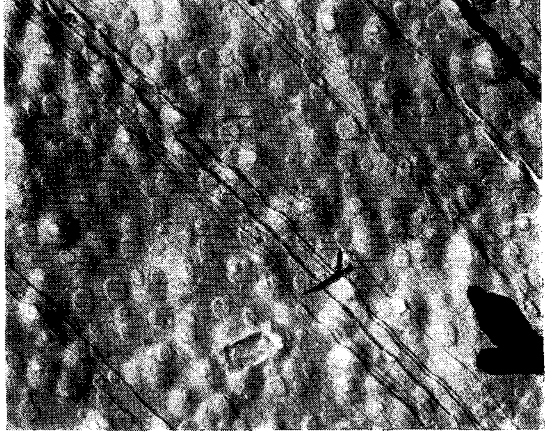


Photo 16. Aged for 64 hr at 870°C, reduced 20%, ×19,000.

Photo 17. Aged for 64 hr at 870°C, reduced 20%, ×19,000.

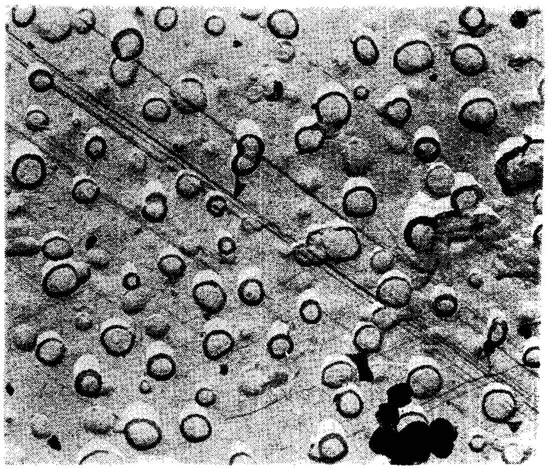


Photo 18. Aged for 125 hr at 870°C, reduced at the yield stress, ×17,500.

Photo 19. Aged for 64 hr at 870°C, reduced 20%, ×9,000.

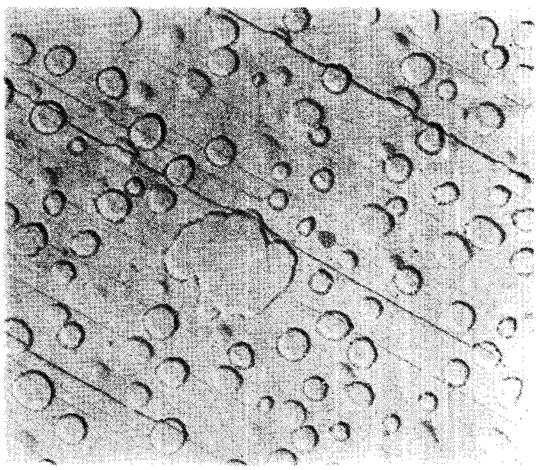


Photo 20. Aged for 9300 hr at 870°C, indented, ×12,000.

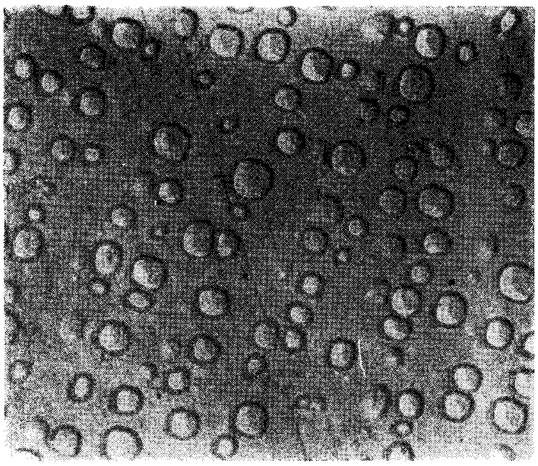


Photo 21. Aged for 9300 hr at 870°C, indented, ×12,000.

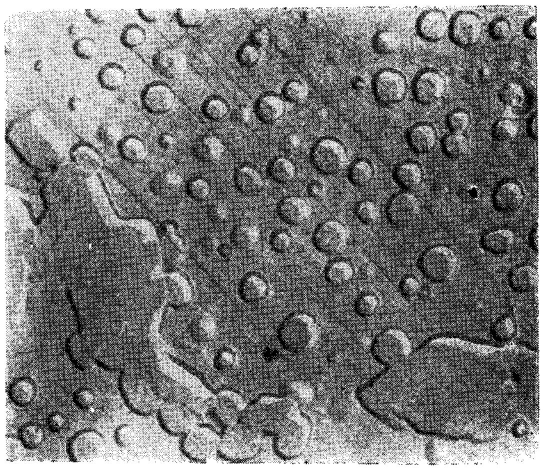


Photo 22. Aged for 9300 hr at 870°C, indented, ×12,000.

Photo 23. Aged for 4 hr at 760°C, reduced 20%, ×4,000.

particles precipitated during heat treatment increased in proportion to the cube-root of aging time.

2) Slip lines occurred on the {111} ⟨110⟩ system and were composed of single slip lines. Slip depth, length of slip lines and number of activated slip systems decreased with increasing hardness.
3) Cross-slip occurred abundantly in all deformed specimens at room temperature, indicating that René 41 has medium or medium to low stacking fault energy.
4) For deformation of 5 to 20% strain, slip lines generally passed through most of γ' particles whose size ranged up to 9000 Å, shearing them with the matrix even after a prolonged heat treatment.
5) The study of the interaction between slip lines and γ' particles led to the conclusion that γ' particles remain at least partially coherent with the matrix even in a greatly overaged condition.

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