SIZE EFFECTS IN THE MECHANICAL PROPERTIES OF SUPERALLOY SINGLE CRYSTALS

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Abstract

The influence of the diameter of [001] axial single crystal samples on the tensile and creep properties of MAR-M002 superalloy at 850 and 900 °C, respectively, has been studied. The tensile results indicate that the load bearing capacities of single crystals increase near the surface. The observed phenomena are interpreted on the basis of the macroscopic model in which the γ-phase is considered to possess fluid-like properties and where, under a [001] directed tensile stress, the γ-phase would flow along pressure gradients within the γ-phase channels between the non-deformable γ' particles. The observation that the load bearing capacity increases near the surface is interpreted to indicate a restriction in the flow of the γ-phase fluid at the surface.

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Introduction

The advent of hollow air-cooled turbine blades, grown in the form of single crystals of nickel-base superalloy, has led to substantial increases in the allowable operating temperatures (1). In such hollow blades there are sections of various thicknesses and it is thus of importance to establish whether section size effects may be present. A study of size effects on stress-rupture and creep properties of CMSX-3 single crystals, has been carried out by Doner and Heckler (1). They found a considerable effect on the stress-rupture properties but only a very small effect on the creep properties.

The main purpose of the present paper is to consider section size effects in the mechanical properties of MAR-M002 single crystals. However, in order to consider the deformation mechanisms involved on a wider basis, studies on the effects of varying sizes of γ' particles as well as the effects of mechanical machining of the gauge length were also carried out.

The results presented in this paper are interpreted generally on the basis of a model proposed by Pollock and Argon (2,3) which considers macroscopic resistance effects to plastic flow. These macroscopic influences result from the flow of the γ-phase "fluid" in between the non-deformable γ' particles. Accordingly it is shown, in the present paper, that a satisfactory explanation of the results on γ' particle sizes and on sample sizes may be arrived at on such a basis. The macroscopic model (2,3) is discussed in detail below.

Background to the Plastic Deformation of Superalloys

The fundamental mechanisms of plastic deformation in nickel-base superalloys have been considered recently by Pollock and Argon (3). For the purpose of the present paper, attention will be confined to their model relating to macroscopic deformation resistance effects. In this regard, and based on an analysis by Schroeder and Webster (4), they considered the γ/γ' composite of the nickel-base superalloy, to consist of non-deforming γ' particles which are held together by the more plastically deformable γ-phase. They argue that once dislocation motion, and hence plastic deformation, occurs within the γ-phase, the latter phase may be viewed as a viscous fluid. Thus when a [001] axial sample is placed under tensile stress, the γ-phase, because of substantial gradients in pressure and negative pressure in channels, will tend to flow from the vertical channels into the horizontal channels between the γ' particles. This proposed phenomenon is depicted in an exaggerated manner in Figure 1. According to Pollock and Argon (3), the negative pressures over the horizontal γ channels would offer considerable load support during the creep of single crystal specimens when these are pulled in the [001] direction.

In the opinion of the present authors, the viscosity of the γ-fluid would be influenced by the density of mobile dislocations. Since a low density would lead to increased resistance to flow, it follows that such a low density may be associated with a high viscosity of the γ-fluid and vice versa. A satisfactory interpretation of the present results was found to be possible by employing these concepts.

In the present paper, results on deformation near the surface of the sample need to be considered. For this purpose the right hand edge (say) of the sample in Figure 1 may be considered to be a free surface, whereas the other edge is considered to be within the interior of the sample. It appears likely that the flow of the γ-phase "fluid" would occur under some considerable restriction at such a free surface, and that a singularity in the flux may exist there. Accordingly, it is possible that the γ-phase fluid in horizontal channels which end at

the surface, would assume a concave shape at that surface. The formation of such a sharp curvature within the γ-phase, would require appropriate dislocation translation within the material and it is likely that the γ-phase, there, would harden considerably because of resulting dislocation interactions. Thus it could be envisaged that the density of mobile dislocations would decrease near the surface and thus that the viscosity of the γ-fluid would increase there.

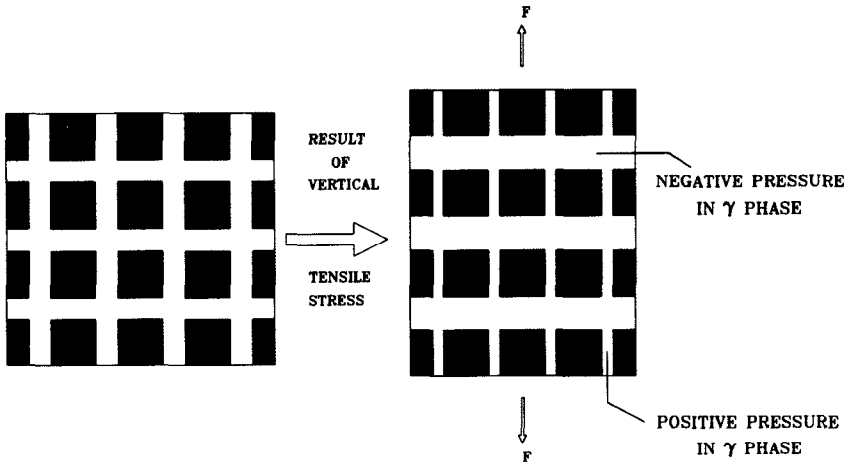

Figure 1 - Diagrammatic representation of the γ-fluid model.

The present experimental results, which indicate an increased load-bearing capacity near the surface, will be examined in terms of the macroscopic model discussed above.

Experimental

The single crystals of MAR-M002 alloy, with a composition as given below, were prepared in two batches by standard casting methods. Subsequently, the crystals were solution heat treated in vacuum at 1225 °C and quenched. Thereafter each batch was subjected to differing ageing cycles, to obtain, in one instance, a γ' particle size of 0.65 and in the other 0.28 μm.

Table I Chemical Composition (wt.%) of MAR-M002

Alloy	Ni	Cr	Co	W	Ta	Al	Ti	Zr	Hf	C
MAR-M002	60	9	10	10	2.5	5.5	1.5	0.05	1.5	0.14

The cylindrical gauge lengths of single crystals utilized in tensile tests, were machined in a specially designed lathe, in which surface removal could be effected by means of an electrolytic jet device. Hence, completely strain-free machining was possible and gauge diameters down to 0.5 mm were successfully prepared and tested in tension. For example, for the sample shown in Figure 2, the gauge diameter A, was prepared first and subsequently tested, while B and C were still of the original diameter which was greater than that of A. Thus there was no deformation of B or C during the tensile test of A. Then B was machined and tested, and then, finally, C. Thus 3 tensile curves could be obtained for different gauge diameters, but for identical composition and microstructure. Most important was the fact that every gauge length was free of extraneous deformation at the start of testing. The tensile testing was done in an argon environment at 850 °C and at a strain rate of 4×10^{-4} s^{-1}.

For creep testing, the heat treatment of the samples was such that the γ' particles had a diameter of about 0.65 μm. The gauge lengths were prepared by the mechanical grinding of cylindrical gauge lengths for diameters of 4.0 mm down to 1.4 mm. The creep testing was done at 900 °C at a stress of 300 MPa.

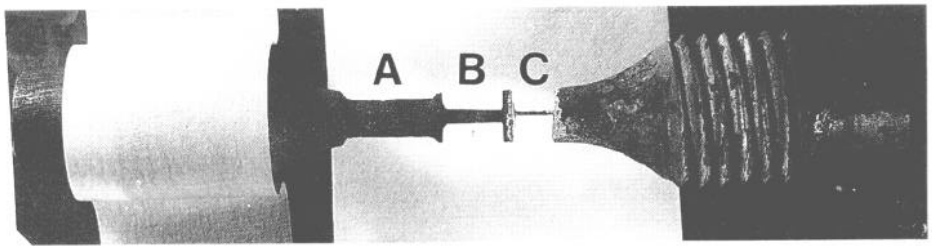

Figure 2 - Three electrolytically machined gauge lengths of different diameter on a single sample.

Results

Experiments on MAR-M002 Single Crystals

In Figure 3 are shown two curves which demonstrate the pronounced effect which different sizes of γ' particles may have on the plastic stress-strain curve in tension. The crystal for curve A contained γ' particles with an average size of 0.65 μm, whereas for curve B, the average size was 0.28 μm. Both crystals yielded at about 700 MPa. For curve A, the stress rises smoothly during a small amount of plastic strain until a stress of about 820 MPa is reached. At this point the deformation becomes plastically unstable and continuing deformation would lead to necking and fracture. For curve B, the stress rises smoothly with strain until about 730 MPa, at which point a massive yield drop in stress is observed. The yield drop bottoms out at about 660 MPa and then, after a transition stage, the rise in stress with strain follows a distinctly linear relationship for a considerable stress interval. At the end of this interval, the stress-strain relationship becomes parabolic, leading to plastic instability and fracture.

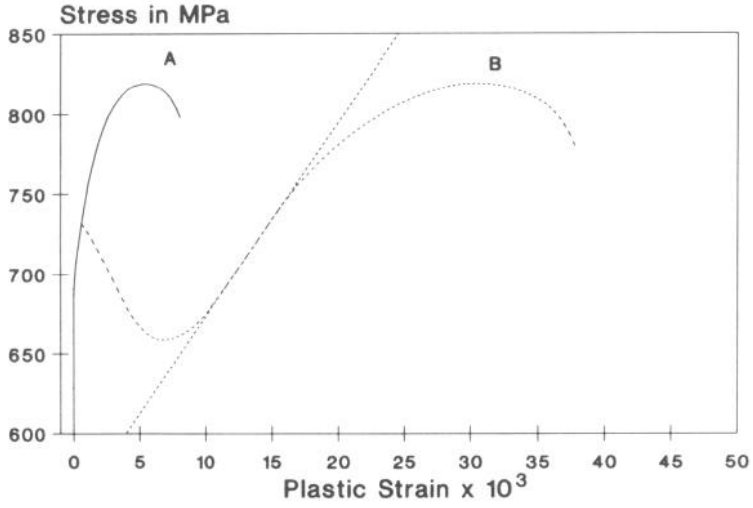

Figure 3 - Stress-strain curves of [001]-axial single crystals of MAR-M002 tested at 850 °C where the γ' sizes of A and B are 0.65 and 0.28 μm, respectively.

In Figure 4 are shown two curves which demonstrate the very significant effect that mechanical grinding of a gauge length may have on the mechanical properties of single crystals. The crystals A and B were from the same batch of casting and had been heat treated simultaneously under identical conditions, leading to a γ'-size of 0.65 μm. The gauge length of crystal A was machined electrolytically, whereas that of B was machined mechanically. It is clear from the shape of curve A, that there is no yield point after plastic flow begins. The plastic-stress-strain relationship is essentially parabolic to the point where plastic instability sets in. For curve B, on the other hand, there is a measure of yield drop after some considerable plastic flow has occurred. This is followed by a distinctly linear region of relatively small slope. Further, the total plastic strain extent of B, before plastic instability set in, appears to have been increased by a factor of about 5 by the mechanical machining.

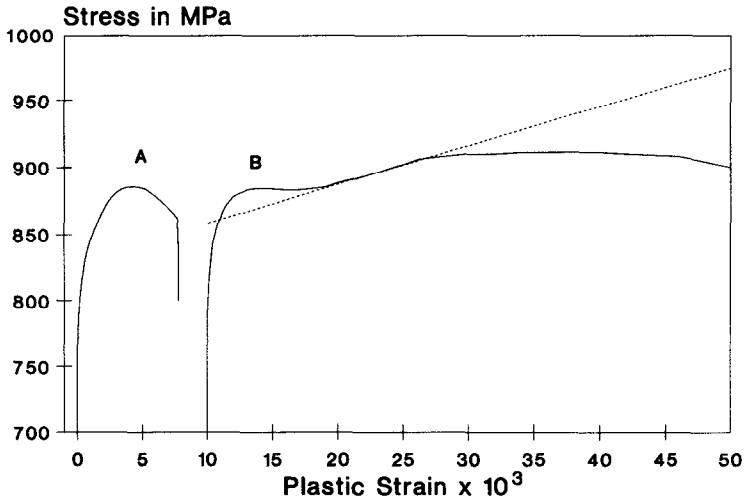

Figure 4 - The stress strain curves of two [001]-axial MAR-M002 single crystals,with gauge lengths A and B, respectively, machined electrolytically and mechanically. The testing temperature was 850 °C.

In Figure 5 are represented the three stress-strain curves A, B and C which were obtained, correspondingly, from electrolytically machined gauge lengths A, B and C of the tensile sample shown in Figure 2. The gauge diameters of A, B and C were 4.9, 1.8 and 0.5 mm, respectively. The microstructure, which was identical for the three gauge lengths, was examined by transmission electron microscopy, and found to have an average γ' particle size of 0.28 μm. It is clear, from Figure 5, that there is a consistent variation in the stress-strain curves in relation to the gauge diameters. Firstly, the yield drop is greatest for the largest diameter sample. Secondly, the upper yield point is the highest for the smallest diameter sample. Thirdly, the rate of work-hardening within the linear region of the curve (slope shown by broken lines) is also the highest for the smallest diameter sample. Fourthly, the extents of stress and of strain of the linear region is the least for the smallest diameter. Finally, the extent of strain, following yield, to the point where a linear stress-strain relationship begins, is the least for the smallest diameter sample.

In Figure 6, the minimum creep rate in MAR-M002 single crystals containing 0.65 μm sized γ' particles, in which the gauge lengths were mechanically machined, is plotted as a function of the gauge diameter. The starting stress was 300 MPa and the temperature 900 °C. It appears that a consistent relationship between the minimum creep rate and the diameter

cannot be deduced from these results. This inconsistency is probably related to mechanical damage during the grinding of the gauge diameters, as will be discussed. Measurements on the creep-rupture life as a function of diameter displayed the same degree of inconsistency.

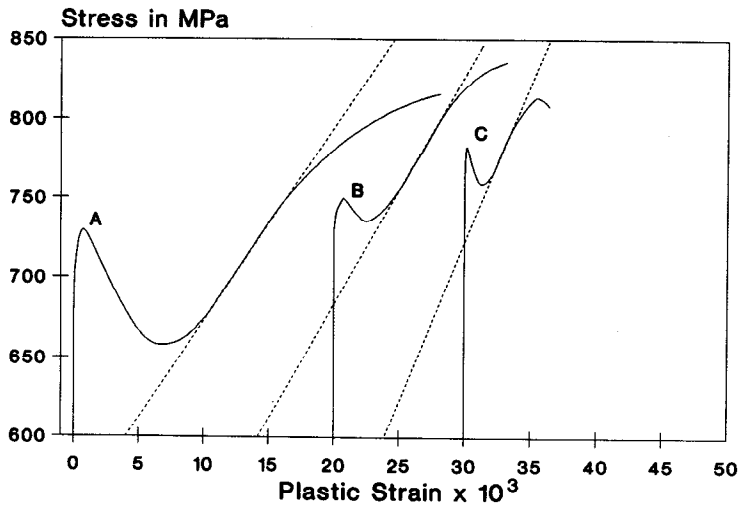

Figure 5 - Stress-strain curves of gauge lengths A, B and C as shown in Figure 2 with gauge diameters 4.9, 1.8 and 0.5 mm, respectively as measured at 850 °C. The gauge lengths were machined electrolytically.

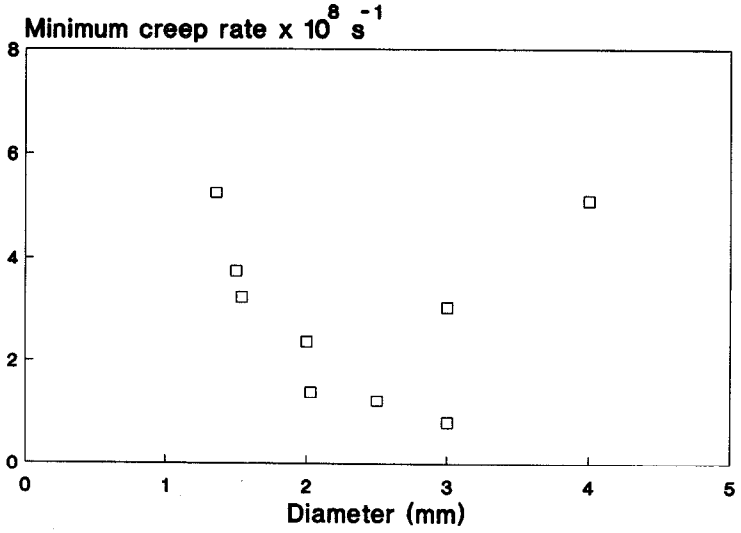

Figure 6 - The minimum creep rate in [001]-axial MAR-M002 single crystals of various gauge diameters, as measured at 900 °C and a stress of 300 MPa. The gauge lengths were machined mechanically.

Discussion

The phenomenon in Figure 3, where a distinct difference in plastic tensile behaviour is manifested by single crystals of the same alloy, each containing a different size γ' particles, may be understood as follows. In terms of the γ-fluid concept of Pollock and Argon (4), it is likely that curve A (γ'-size 0.65 μm) represents the flow of a very viscous fluid. This fluid is required to flow from the vertical channels (Figure 1) into the horizontal channels.

The fluid evidently maintains its high viscosity throughout the tensile test, probably because no meaningful dislocation multiplication is initiated at any stage. Thus the stress at which γ' particles are penetrated by dislocations, was reached before significant dislocation multiplication could occur in the γ-phase. It follows that plastic instability and fracture occurred at a low strain for materials of this nature, at the temperature and strain rate of the test.

These deductions are supported by the result in Figure 4, where the curve B is representative of a single crystal subjected to mechanical grinding. Such grinding probably introduces a high density of mobile dislocations into the γ-phase within a radial region approximately 0.5 mm deep which bounds the surface that had been subjected to grinding. These mobile dislocations would effectively lower the viscosity of the γ-fluid and bring about the rather complex curve B, which, in contrast to curve A (electrolytically machined), now has a linear region, but probably represents the stress-strain characteristics of a non-uniform material.

Returning to Figure 3, the curve B (γ'-size 0.28 μm), shows by way of the massive yield drop, that very intense multiplication of mobile dislocations occurred after yielding. Clearly there must be a reason why this dislocation multiplication should be related to the γ' particle size. Now, Pollock and Argon (3), by finite element analysis, showed that for the tensile axis along [001], the highest stresses in the γ-phase occurred (after plastic strain of 0.006) near each corner of the cuboidal γ' particle. These corners, then, would be the most likely sites for dislocation multiplication. Now, when the size of γ' particles is reduced from D_A to D_B, it follows, from elementary geometry, that the density of cube corners will increase by the factor $(D_A/D_B)^3$. Thus for $D_A = 0.65$ and $D_B = 0.28$ μm, this factor becomes about 12.5. It follows, therefore, that the probability of a high rate of dislocation multiplication for sample B after yield, would be much more favourable than for sample A. Caron, Ohta, Nakagawa and Khan (5) observed a related effect, of the influence of the size of γ' particles on the creep curves of [001]-axial CMSX-2 single crystals. They interpreted their results as indicating an extensive cooperative shearing of the γ/γ' structure for γ' particles in the 0,22 μm range. However, for the present tensile results, a more consistent interpretation of the results, seen as a whole, can be found via the macroscopic model of the resistance to flow of the γ-phase. The results are, therefore, considered on that basis.

The massive yield drop in curve B may be interpreted in terms of the γ-fluid concept by coupling the viscosity of the fluid with the density of mobile dislocations. Thus after yielding (in curve B), the mobile dislocation density increases, leading to a decrease in the viscosity of the fluid, which reaches a minimum at the lower yield point. Then, after a short transitional rise, the viscosity begins to increase linearly with strain, corresponding to the linear region of the curve. Obviously, suitable dislocation interaction mechanisms would be required to bring about the necessary rate of increase in the viscosity of the fluid so as to produce a linear region in the stress-strain curve.

The effects of sample diameter on tensile properties as shown in Figure 5, may also be related to the γ-fluid concept. It is proposed, with reference to Figure 1, that the flow of the γ-fluid is in some way restricted near the surface, since there would be no fluid reservoir on the argon environmental side of the surface. The results in Figure 5, in terms of such a model, would suggest that the load bearing capacity of the material near the surface is considerably greater than within the interior. This follows from the fact that the yield point is higher for C than for A. The fact that the extent of strain, after yield, up to the linear part of the curve, is much smaller for C than for A, suggests that, at the surface, dislocation multiplication does occur, but that accompanying dislocation interaction is more intensive

there than in the interior. Thus the rate of decrease in the density of mobile dislocations is greater than in the interior. It follows that the viscosity of the γ-fluid would rise more rapidly at the surface. On this basis, the observation that the slope of the linear region in curve C is greater than that in curve A, is consistent with the γ-fluid model.

The inconsistency of the creep results in Figure 6, emphasize the very significant perturbing influence that the mechanical machining of gauge lengths may have on measurements where the mobile dislocation density is important in the behaviour of the material. This fact has already been definitively illustrated and discussed, with reference to Figure 4, in the present paper. For the case, as in Figure 6, where different diameters of sample were produced by mechanical machining, the deleterious effects are probably exacerbated for the following reasons. It was suggested earlier, that the zone of damaged material extends for about 0.5 mm below the machined surface. Thus, for the situation as in Figure 6, where the maximum and minimum diameters of 4 and 1.4 mm apply, the ratios of the volume of damaged material to the total volume are 43 and 92%, respectively. Clearly, then, this ratio will change drastically with decreasing diameter. Thus creep tests on such samples would probably provide spurious results. This aspect is reflected by the results shown in Figure 6.

It follows, that to obtain relevant results about the size effect in creep, it will be essential to carry out experiments which are modelled on the tensile experiments in Figure 5, with the same preparation method as illustrated in Figure 2.

The tensile results in Figure 5 permit a preliminary prediction on the size effect in creep, however. As mentioned earlier, the linear parts of the curves show a strain extent which is proportional to the diameter of the sample. It is believed that the extent of this region of stable flow would provide a measure of the expected creep-rupture life of the sample. Thus it may be predicted from the results in Figure 5 that the creep-rupture life would decrease substantially with decreasing sample diameter, especially in the region below 2 mm diameter. This prediction is essentially in agreement with published creep results on single crystal samples of PWA 1480 alloy (6).

Summary and Conclusions

Tensile tests conducted at 850 °C on two batches of [001]-axial single crystals of MAR-M002 superalloy, containing in the one instance 0.28 and in the other 0.65 μm sized γ' particles, are described. It is shown that a consistent interpretation of the different modes of tensile behaviour of the two microstructures is possible in terms of a model based on macroscopic deformation resistance effects. This model involves the flow of the γ-phase fluid within the channels between non-deforming γ' particles.

The sensitivity of tensile tests to mechanical damage in the grinding of gauge lengths is illustrated by comparing the tensile curves of two identical samples prepared firstly by a strain free electrolytic method and, secondly, by mechanical grinding. A drastic change in the tensile deformation characteristics is observed as a result of mechanical grinding. It is believed that, in the present investigation, this phenomenon was responsible for the fact that creep measurements on samples of different diameter, showed an inconsistent variation as a function of sample size.

In crystals of identical microstructure but with varying gauge diameter, where the gauge lengths were prepared by strain-free electrolytic jet machining, tensile measurements indicate that the load bearing capacity increases with decreasing diameter in [001]-axial single crystals

of MAR-M002 alloy. This result is interpreted in terms of the flow of the γ-phase "fluid" in the channels between γ' particles near the surface. It is argued that, at the surface, the restriction in flow of the γ-phase may cause a reduction in the mobile dislocation density. This again, may cause an effective increase in flow stress near the surface. Predictions about the creep-rupture life of samples of varying diameter, based on the latter tensile results, are shown to be in agreement with creep results in the literature.

Acknowledgements

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