ATTRACTIVE DISLOCATION AND PARTICLE INTERACTIONS IN ODS SUPERALLOYS --

AND IMPLICATIONS

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Summary

For the oxide dispersion strengthened (ODS) alloys, dislocation-particle interaction is generally believed to be repulsive since the elastic constants of the oxide dispersoids are believed to be greater than those of the matrix. High temperature strengthening is thought of in terms of a dislocation climb modified Orowan type bowing mechanism between the particles. Dislocation escape occurs when the stress is sufficient to cause the dislocation to bow to a critical configuration. Contrary to this general belief, through detailed TEM microscopy study of crept and hot tensile tested Inconel MA 754, we have found and will report in this paper that the interaction between dislocation and oxide dispersoid is in fact attractive, with the high temperature properties of ODS alloys stemming from this unexpected attractive interaction. In particular, the characteristic and important threshold behavior of ODS alloys during creep at high temperatures is discussed. It is proposed that the finding of attractive interaction opens new avenues for the alloy design of ODS alloys, including the pinning of dislocations next to the dispersoids by additional elements and the purposeful introduction of non-adherent interfaces between dispersoids and matrix. In the limit, the high temperature (or low strain rate) threshold stress of, say, Inconel MA 754 at 1093 deg. C, can be increased by a factor of two from 94 MPa (14 ksi) to 188 MPa (28 ksi).

Introduction

Dislocation-particle interactions are of fundamental importance in understanding the deformation behavior of particle strengthened systems. For oxide dispersion strengthened (ODS) mechanically alloyed (MA) alloys, the fine oxide particles are plastically nondeformable. Shearing of the particles by mobile dislocations is then not a viable mode of deformation as is the case with the gamma prime precipitation strengthened superalloys.

Heretofore it is believed that nonshearable particles strengthen through either the classical Orowan mechanism or the Orowan mechanism modified by climb of the dislocations over the obstacles (1,2). Implicit in the Orowan mechanism is that the elastic moduli of the obstacles is higher than that of the metallic matrix, resulting in dislocation and particle repulsion and in the classical Orowan encounter (1) of mobile dislocation bowing between two particles, reaching (in the most simplistic view) the critical configuration of a complete half-loop before becoming unstable, folding back on itself and escaping by leaving an Orowan loop around each particle. Figure 10.1 in reference 3 is a typical TEM representation of the Orowan mechanism caught in actual operation (3) in a system where the elastic moduli of the oxide particle are higher than those of the matrix. At high temperatures, when dislocation climb can occur, it has been shown that now as the mobile dislocation bows, it is also climbing over the particles and by the time it reaches the other side and escapes, the bow will not yet reach the half-loop configuration (2). Since the resisting stress is a direct function of dislocation line tension, which in turn is dependent upon how bowed the mobile dislocation becomes before escape, the strengthening stress in ODS, MA alloys has been taken to be between the Orowan stress and a high temperature (or low strain rate) limiting value of about 0.4 (2) of the appropriate Orowan type stress. Accordingly, traditional thinking would have it that the limiting or threshold strength for ODS-MA alloys at high temperatures would be about 0.4 of the Orowan stress. At first glance this appears to be the case.

A representative example of the high temperature behavior of ODS-MA alloys is given in Fig. 1 for single crystal Ni20Cr2ThO_2 (4) and for high grain aspect ratio Inconel MA 754 (5,6). Grain boundary effects can be excluded for these systems, so that their high temperature, slow strain rate (i.e. creep) behavior can indeed be attributed to dislocation particle interaction. As shown in Figure 1, ODS alloys possess a characteristic "threshold stress" for dislocation creep, below which no detectable creep occurs. From temperature and modulus compensated yield strength data, Pharr and Nix (7) determined that the modulus compensated Orowan stress for the Lund and Nix system, Fig. 1a, is about 1.7 times greater than the threshold shown in the figure. Similarly, Nardone et al. (6) concluded that the modulus compensated Orowan stress for MA 754, Fig. 1b, is about twice that of the threshold shown in that figure. Petkovic-Luton et al. concluded that for the Inconel MA 956 system that the Orowan stress is also about a factor of two greater than the experimentally determined threshold stress for that ODS-MA alloy (8).

Accordingly, it does appear that a climb modified Orowan mechanism may determine the limited high temperature creep strength of ODS-MA alloys. In other words, the high temperature threshold strength of an ODS alloy will always be less than about one-half the temperature compensated Orowan stress produced by the interparticle spacings and other geometric features of that alloy. We will show below that this is a false constraint, since the dislocation dispersoid interaction can be an attractive one, and the

Figure 1 - The diffusion compensated strain rate versus the modulus compensated applied stress for a) Ni-20Cr-2ThO₂ single crystals (data from [4]) and b) Inconel MA 754 (open data points from [5] and solid data points from this study). See text for discussion of dotted curve.

threshold may be increased by a factor of two through appropriate microchemistry and oxide/matrix interface modifications.

Experimental

The alloy chosen for this study was Inconel MA 754 which is essentially a Ni20Cr solid solution strengthened by Y_2O_3 dispersoids; a detailed microstructural characterization has been reported elsewhere by Howson et al. (5). The grain aspect ratio is about 10. The Y_2O_3 fraction is 2.5 percent by volume. The average oxide size is 14 nm, and interparticle spacing was determined to be 110 nm (5). In order to confirm earlier (5) strain rate versus stress data, and to produce structures for TEM analysis, creep tests were done on a dead-load machine and the reported creep rates are ± 5 percent in accuracy (5). Temperature was maintained to ± 2 deg. C. In order to obtain higher strain rate data tensile tests were also performed in a computer-controlled MTS machine under constant true strain rate conditions. All mechanical tests were done with specimen axis parallel to the long grain axis to eliminate grain boundary effects during creep.

The representative TEM microstructures investigated were of five specimens tested in the disk (760 deg. C) temperature regime and four specimens tested in the vane or blade (1093 deg. C) temperature regime. About half of the tests were interrupted at the saturation stress (9) under tensile conditions and half in the steady state regime under creep loading conditions. Some specimens were cooled on load by opening the split furnaces. In addition some specimens were crept into steady state, unloaded and allowed to anneal at temperature for one to three hours to determine the lower energy configurations of the dislocations at the particles. TEM foils were prepared parallel to the primary (111) slip plane in most cases. Foils were thinned with a 55% ethanol, 30% n-Butanol and 15% perchloric acid (70%) solution and under an applied voltage of 40V at -6 deg. C. All TEM observations were made on a JEOL Model JEM-100CX scanning-transmission electron microscope. Observations of dislocation-particle interaction were usually made over several specimen stage rotations, with tilts as high as 50 degrees from the original electron beam and foil orientation.

Results

The creep and tensile data obtained from specimens from which our TEM microstructural observations were made, as well as the data of Howson et al. on the same material heat (5), are plotted in Fig. 1b. All data points appear to be consistent with each other, and, as mentioned, the diffusion compensated strain rate versus modulus compensated stress plot shows a characteristic threshold stress below which no detectable creep occurs. The plot shows a very high stress exponent in the low stress regime which tends to lessen toward the higher modulus compensated stresses. In producing the plot, the diffusion and modulus values were taken from those discussed in the earlier paper on this subject (6).

The fine microstructural observations consistently showed the dislocations to be pinned at particles. Figure 2 includes representative TEM views from specimens crept and cooled on load at 760 deg. C and 1093 deg. C, respectfully. Note the dispersoid free zones are void of dislocations. Figure 3 shows typical sequences of views on specific dislocation-particle interactions at various stage tilts, revealing that dislocations maintain intimate contact with the particles though the dislocation appears to be on the departure side of the strengthening dispersoids. Such intimate

Figure 2 - Representative TEM micrograph of MA 754 deformed and cooled on load a) after tensile testing at 1093 deg. C ($\dot{\epsilon} = 1 \times 10^{-4}/s$ and $\epsilon = .02$) and b) crept to steady state at 760 deg. C ($\dot{\epsilon} = 9 \times 10^{-8}/s$ and $\epsilon = .02$), showing dislocations attractively pinned to the fine oxide particles.

Figure 3 - Representative TEM micrographs of dislocations and specific particle interactions at a variety of specimen stage tilts; specimens were crept to steady state at 1093 deg. C ($\dot{\epsilon} = 1.28 \times 10^{-3}/s$ and $\epsilon = 0.01$) and cooled on load a) 0 deg. tilt, b) 20 deg., and c) 45 deg.; crept at 760 deg. C ($\dot{\epsilon} = 2.15 \times 10^{-5}/s$ and $\epsilon = .02$) and cooled on load d) 0 deg. tilt, e) 20 deg. and f) 50 deg. Note in both cases the dislocations are pinned on particles' departure sides, as evidenced by the fact that the dislocations are stuck on the sides of the particles from which the dislocations are bowing away.

configurations were observed in thirty other foils from specimens tested. These observations are consistent with the existence of an attractive dislocation-particle interaction.

To further confirm the existence of an attractive interaction, dislocation configurations in specimens crept to steady state and then annealed off load were investigated. The dislocations still remained firmly attached to the particles, Figs. 4 and 5, even after loads were removed at the test temperature.

We observed an absence of any detectable Orowan loops around particles. The fact that no looping is seen does not in itself rule out repulsive interactions between dislocations and the oxide particles. Brown and Stobbs (3) have shown that the relaxation of loops around dispersoids could also account for their absence. The observed intimacy between dislocations and particles on the departure side of the particles, even after long time, off-load annealing, is indisputable evidence of attractive interactions, however.

Discussion

Although the implications of attractive interactions between dislocations and oxide particles were not then recognized, that dislocations in ODS alloys may be stuck to the oxide particles are discernible in electron micrographs appearing in past ODS related papers. TEM micrographs of TD-nickel and TD-nichrome show such evidence in papers published as early as 1966. (10,11) The comprehensive paper published in 1980 (5) on Inconel MA 754 also contained micrographs showing strong residual pinning of dislocations to the oxide particles, much like those in the representative micrographs in this paper. The first proposed connection between creep thresholds and the observed strong attractive interaction of dislocations and oxide particles was made very recently, and independently, by Nardone and Tien (12) and by Srolovitz, et al. (13). The former paper (12) was accompanied by supporting TEM micrographs. Consequently, controversies did arise as to whether the dislocations were indeed stuck to the particles resulting in pinning even on the departure side of the particles. Arguments were made that the dislocations may instead be lying on planes above or below the particles and pinned by either the foil's free surfaces or sessile dislocations perpendicular to the electron view. Accordingly, the result from our careful and effort consuming tilting experiments are invaluable, since they represent indisputable evidence that there can be a strong attractive interaction between the mobile dislocations and the strengthening obstacles (the oxide particles). A dislocation line not really in contact with particles, may visually appear to be in contact in one view, but this illusion cannot be sustained through view tilting of over 45 degrees.

The reason for an attractive dislocation particle interaction must stem from a decrease in the energy of the system as a result of dislocation/particle association. The factors that can account for this decrease in energy are 1) a change in the line and core energy of the dislocation due to elastic modulus differences and 2) microchemistry effects at the oxide interface.

As mentioned, superficially, a decrease in the elastic energy of the dislocation seems unlikely since the polycrystalline elastic modulus of the oxide particles (Y_2O_3) exceeds that of the fcc nickel-chromium matrix. Under these conditions, elasticity theory shows that a repulsive interaction should exist between the dislocations and the particles. The elastic

Figure 4 - TEM micrographs of MA 754 crept to steady state, unloaded and annealed, to allow dislocations to rearrange to lower energy configurations; a) 1093 deg. C and b) 760 deg. C. Note that the dislocations still maintain close contact with the dispersoids.

Figure 5 - TEM micrographs of MA 754 of dislocation-particle configurations at a variety of tilts; specimens were crept and then annealed off load at 1093 deg. C a) 0 deg. tilt, b) 20 deg. and c) 45 deg.; and at 760 deg. C d) 0 deg. tilt and e) 25 deg.. Note the dislocations still maintaining contact with the particles at all tilts shown.

constants are very anisotropic in nature, however. For example, the 760 deg. C Young's modulus for textured MA 754 being 110×10^3 MPa, while the untextured material has a 760 deg. C Young's modulus of 154×10^3 MPa. (14) This confirms that there is a wide range of values for the elastic constants in a grain of Inconel MA 754. In addition, the oxides are essentially small single crystals, also having anisotropic elastic constants. At 760 deg. C, the Young's modulus for polycrystalline Y_2O_3 was determined to be 145×10^3 MPa. (15) Accordingly, there can be a wide range of relative orientations between oxide and matrix that can allow for an attractive dislocation-particle interaction, due to a decrease in the elastic energy of the dislocation.

The limit in attractive interaction is that between dislocations and voids, since such encounters result in the elimination of a segment of the dislocation line (and thus core and elastic energy) equal to the diameter of the void. It stands to reason then, regardless of the sign of the modulus difference between oxide particles and matrices, a maximum attractive interaction will occur if at temperature the oxide-matrix interface becomes non-adherent, separates and forms a localized minute void space - resulting effectively in a zero modulus particle. This idealized and hoped for extreme is not occurring in MA 754, however. As shown in the tilting sequence views in Figs. 3 and 5, dislocations that seem to have disappeared at particles were in fact pinned to the departure side of the oxide particles.

The exact nature of this pinning is not known at this time. (6) Certainly, trace impurities (H, O, N, C, S, etc.) in alloys will segregate and concentrate at the oxide/matrix interface. These or other impurities can then transfer on to the dislocation that is in intimate contact with the interface due to the afore-discussed and observed attraction between oxide particle and dislocation. Once stuck on the dislocations the impurities can pin the dislocation on the departure side of the dispersoids either through Cottrell-drag or by the lowering of the localized energy of the dislocation line segment that is wrapped about the particle. Dislocation escape then occurs by the applied stress bowing the dislocation which is now stuck at the departure sides of, say, two adjacent particles, until the line tension force exceeds the pinning forces due to the lower (localized) energy of the dislocation segment at the particles.

A detailed model for overcoming attractive dislocation-particle interaction has been derived (6), and the calculations show that a modest reduction of the stuck segment energy by only 5 percent is necessary in order to result in the threshold values of about 0.5 of the Orowan stress. Indeed, in the limit, for 100% reduction, i.e., in the wished-for non-adherent oxide/matrix interface case, it can be easily shown that the threshold value will then increase to be equal to the Orowan stress. Accordingly, attractive dislocation-oxide particle interaction precludes the limitation of a climb-modified lowering of the Orowan stress as the high temperature threshold strength. This means that the threshold in Fig. 1b, which corresponds to a stress of 94 MPa at 1093 deg. C, can become as high as 188 MPa. Please note that a factor of two increase in threshold strength will result not only in a doubling of the fail-safe applied design stress, but will also result in a shift of the temperature normalized creep strain rate curve to the right (see dotted curve). As can be seen, Fig. 1b, such a shift will result in at least a two-orders-of-magnitude lowering of creep rates at applied stresses above the threshold.

Concluding Remarks

The detailed TEM results of this study show beyond a reasonable doubt that the strengthening mechanism in such ODS, MA alloys as Inconel MA 754 is one of overcoming the attractive dislocation-oxide particle interaction. This type of strengthening is significantly different than the classical Orowan-type mechanism, which is based on repulsive dislocation-oxide particle interaction. Accordingly, the high temperature strength and creep resistance of the ODS alloys can be altered and improved by altering not only interparticle spacing (particle size and volume fraction), but also by manipulating the adherence of the dispersoid/matrix interface, and by local chemistries. Indeed, we show that the results strongly suggest that the infinite-life threshold creep strength can be significantly raised and the creep rates at higher stresses lowered by orders-of-magnitude if the interface is made non-adherent resulting in effect in a circumferential void space.

How this can be accomplished requires, of course, further research into adsorbates, expansion coefficients, interfacial strength, and especially the matrix and oxide bonding steps during mechanical alloying. The results of this paper just underscores the need, and define the potential gains if it can be accomplished.

Acknowledgements

We thank the Materials Science Division of the NSF for supporting this work under Grant DMR 80-11402. Dr. Robert Reynik was the Program Manager of this Grant. Inconel MA is an alloy designation of the International Nickel Family of Companies. We wish also to thank INCO for fellowship support of Ms. Cooper.

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