Tensile properties of mechanically alloyed oxide dispersion strengthened iron alloys Part 2 – Physical interpretation of yield strength

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The components of the yield strength of MA956, a mechanically alloyed oxide dispersion strengthened iron base superalloy, have been investigated quantitatively. It is found that much of the difference in strength between the recrystallised and unrecrystallised forms can be explained in terms of the grain structure. The contribution from dispersion strengthening has been estimated using dislocation theory and has been demonstrated to be consistent with that measured experimentally. The temperature dependence of the yield strength has also been studied; some of the effects observed in the range 500–600°C can be attributed to the change in the intrinsic strength of pure annealed iron. MST/3914

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Introduction

In Part 1 of the present paper[1] a quantitative empirical analysis of the mechanical properties of mechanically alloyed (MA) iron base oxide dispersion strengthened (ODS) alloys as a function of a large number of variables was presented. These included virtually all the parameters which are known to affect the properties, such as the detailed chemical composition (Cr, Al, Ti, Mo, yttria), any recrystallisation or aging treatment, the extent of cold work, the test temperature, and the strain rate. The method involved neural network analysis which produced models which are non-linear and incorporate interactions between variables. Such models are extremely useful in alloy design and data interpretation, but any model which includes a large number of interacting variables becomes difficult to appreciate using the principles of physical metallurgy. This is because the latter tend to be simple in the context of sophisticated industrial alloys, but give insight and are easier to picture.

Part 1 of the present paper[1] dealt with the yield strength, ultimate tensile strength, and elongation. Of these, the yield strength should be the best behaved mechanical property given that the other two rely on complex phenomena related to large degrees of homogeneous and inhomogeneous deformation. The purpose of the present work was, therefore, to attempt a better physical understanding of the yield strength of these most unusual alloys, the details of which can be found in Part 1. However, a brief introduction is presented below for the sake of completeness.

Mechanically alloyed iron base oxide dispersion strengthened alloys are made by ball milling an appropriate blend of metallic powders and oxide until an intimate mixture or solid solution is formed. This alloy is then consolidated by extrusion and high temperature rolling deformation to produce bars or sheets which have a deformed microstructure with submicrometre width grains elongated along the working direction. The dislocation density has been reported to be about $10^{15}\ m^{-2}$ (Ref. 2). The alloys are too hard in this condition and so are heat treated after consolidation. The heat treatment leads to recrystallisation into a coarse and elongated grained microstructure which is responsible for the high creep deformation resistance of the alloys.

The MA956 alloy, which is the focus of the present work, is a ferritic MA-ODS steel with the nominal composition Fe–20Cr–4·5Al–0·5Ti–0·5Y_2O_3(wt-%). It is strengthened against creep by a highly stable fine dispersion of yttrium oxide.[3,4] By conventional standards, this dispersion changes little even as the melting temperature is approached. In the as extruded condition, the alloy has a microstructure with $<1\ \mu m$ grain size and a grain aspect ratio of ~ 30, while in the recrystallised condition the grain size is about 10–50 μm and the aspect ratio is ~ 10 (Ref. 5). The yttria particles added to the starting powders react with aluminium and oxygen from the solid solution to form very fine dispersions of mixed (Y, Al) oxides.[6] The average dispersoid diameter in the as extruded condition is ~ 11 nm.[7]

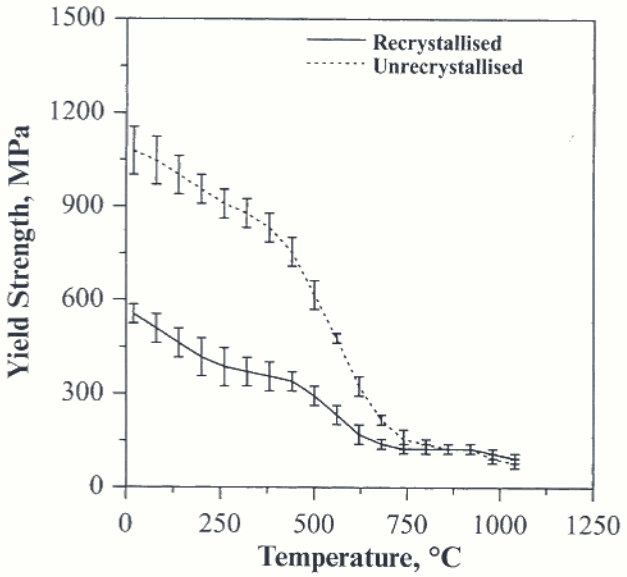
The essential problem which is addressed in the present paper is summarised in Fig. 1 which shows the variation in the yield strength of recrystallised and unrecrystallised MA956 as a function of the temperature. The curves are calculated using the neural network model (Part 1) and therefore represent a best fit empirical interpretation of a large quantity of experimental data with the caveat that overfitting has been avoided as explained in Part 1. The error bars correspond to ± 1 standard deviation and give an indication of the uncertainty in the experimental data as well as the uncertainty in interpreting those data. The major aim of the work presented here is to explain the curves in Fig. 1 on the basis of strengthening theories.

Strength of recrystallised MA956

It is usual to express the yield strength as a linear combination of contributions from a number of mechanisms using the approximation that these mechanisms are essentially non-interacting[8]

$$\sigma_y = \sigma_{Fe} + \sigma_s + \sigma_p + \sigma_g + \sigma_d \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (1)$$

where σ_{Fe} is the strength of the pure annealed matrix, σ_s is solid solution strengthening, σ_p is the particle strengthening, σ_g is the grain boundary strengthening, and σ_d is the dislocation strengthening. The yield strength σ_{Fe} of pure annealed iron at room temperature was calculated as in Ref. 9 with the temperature dependence according to the experimental results by Leslie[10] who studied interstitial free titanium gettered iron. Leslie also reported the temperature dependence of solid solution strengthening by a number of solutes in iron. Although the concentrations he studied did not achieve the levels of interest in the present work, his



1 Neural network predictions for yield strength of recrystallised and unrecrystallised MA956 (Ref. 1)

data for 6 at.-% solute were used to obtain the temperature dependence of solution strengthening for each solute. The absolute values of the room temperature solid solution strengthening were obtained from an empirical expression (where stresses are in MPa) for fully recrystallised ferritic strainless steels (17–25 wt-%Cr) by Lewis and Pickering[11]

$$\sigma_y = 36 + 8{\cdot}5(\text{wt-\%Cr}) + 58(\text{wt-\%Mo}) - 107(\text{wt-\%Ti}) + 15{\cdot}9d^{-1/2} \quad (2a)$$

so that

$$\sigma_s = 8{\cdot}5(\text{wt-\%Cr}) + 58(\text{wt-\%Mo}) - 107(\text{wt-\%Ti}) \quad (2b)$$

The negative coefficient for titanium shows some effect of interstitial solutes.[11] Titanium removes interstitial solutes as TiC or TiN.

Shewfelt and Brown[12] modelled dispersion strengthening as a function of temperature assuming that the dislocations remain on their slip planes except when they are able to overcome the obstacles by climb. Whether or not climb occurs depends on the strain rate and temperature. By comparing theory against experimental results[13] they obtained

$$\sigma_p = \frac{Gb}{\lambda}\left[(0{\cdot}51 \pm 0{\cdot}01) + (0{\cdot}12 \pm 0{\cdot}02) \times \log\left(\frac{\dot{\varepsilon}kTR^2}{4\pi\rho b^2 a_v G\lambda D_0}\right) + (0{\cdot}052 \pm 0{\cdot}009)\left(\frac{Q}{kT}\right)\right] \quad (3)$$

with σ_p in MPa and where G is the shear modulus, b is the Burgers vector, D_0 is the pre-exponential component of the self diffusion coefficient of ferritic iron, Q is the activation energy for this self diffusion coefficient, k is the Boltzmann constant, ρ is the dislocation density, $\dot{\varepsilon}$ is the shear strain rate, R is the particle radius, λ is the square lattice spacing of the particles, and a_v is the area associated with a vacancy. Table 1 shows the values of these parameters for MA956 and the references where the information is from literature. The value for the particle spacing λ has been calculated using an expression by Kelly and Nicholson[14]

$$\lambda = \left(\frac{2\pi}{3}\right)^{1/2}\left(\frac{R^2}{f}\right)^{1/2} \quad (4)$$

where R is the particle radius and f is the volume fraction of particles. The area associated with a vacancy was calculated using $a_v = \pi(b/2)^2$.

Table 1 Parameters for particle strengthening calculation: *a* is lattice parameter of ferrite taken as 0·287 nm

Parameter	Value	Reference
Shear modulus G, GPa	80	15
Burgers vector b, nm	$a\,(3/4)^{1/2} = 0{\cdot}2485$	...
Particle radius R, nm	5·695	7
Dislocation density ρ, m^{-2}	10^{15}	2
D_0 of α-Fe, $m^2\,s^{-1}$	5×10^{-5}	16
Activation energy Q, $J\,mol^{-1}$	240000	16

The calculated components of strength are given in Table 2 and are illustrated as functions of temperature in Fig. 2*a* for recrystallised MA956. At low temperatures the major contributions to the strength are from the intrinsic strength of pure iron and via the dispersoids. However, at temperatures in excess of ~650°C, the dispersoid strengthening becomes dominant. Although the intrinsic resistance to dislocation motion becomes smaller at high temperatures, it never vanishes but reaches a limiting value known as the athermal resistance.[17] The athermal resistance arises from the long range stress fields of obstacles. Fluctuations caused by thermal vibrations are important over distances of the order of a few atoms and hence cannot assist the dislocations to overcome any fields which extend over large distances. This is why the strength of the iron does not tend to zero with increasing temperature (Fig. 2*a*).

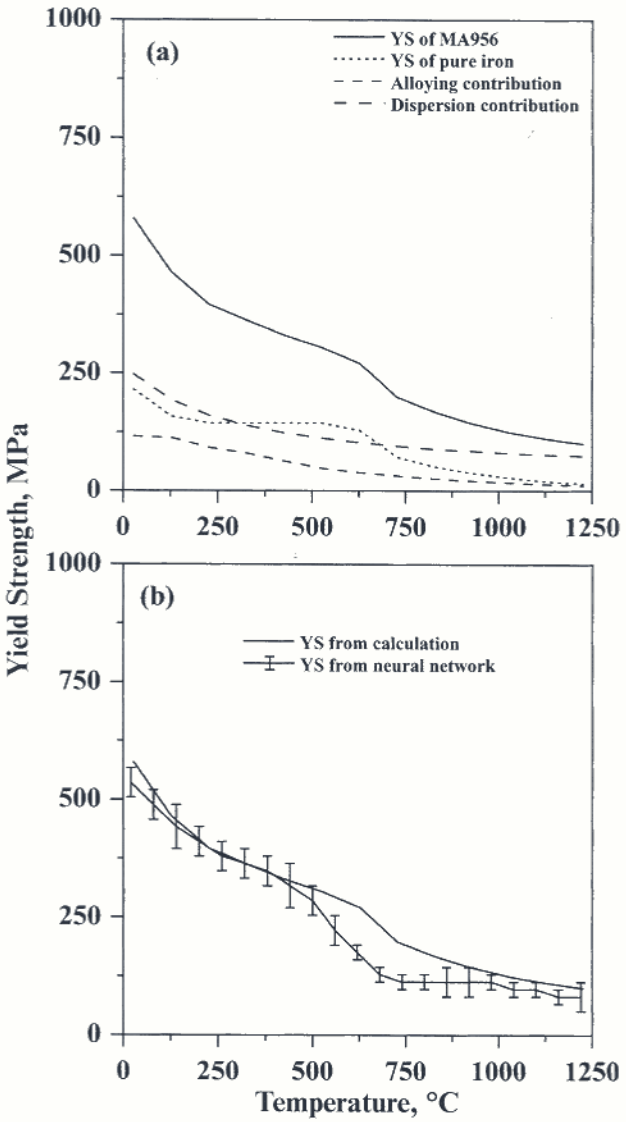
Figure 2*b* shows the comparison of the calculated yield strength with the experimental data as represented by the neural network estimates.[1] The agreement is impressive indicating that the factorisation of strength is reasonable. Furthermore, the accelerating decrease in strength observed in the temperature range 550–650°C is, by comparison with Fig. 2*a*, seen to be a consequence of the similar variation in the strength of the pure iron. Such behaviour is expected because iron atoms become significantly mobile in this temperature range when considered for the slow strain rates typical of tensile tests. There are many other phenomena which reflect the mobility in this temperature range; for example, secondary hardening in alloyed steels.

MEASURED DISPERSOID STRENGTHENING

There might be considerable uncertainties in the estimation of dispersion strengthening. For example, the particles may not be uniformly distributed and are unlikely to have a uniform size.[18,19] To gain more confidence in the analysis, the dispersoids were effectively removed from a recrystallised specimen and the strength measured. This was done by arc melting the alloy in an argon atmosphere, which causes many of the oxides (which have a relatively low

Table 2 Calculated yield strength of recrystallised MA956

Temperature, °C	Yield strength of pure Fe, MPa	Solutes contribution, MPa	Dispersoids contribution, MPa	Total yield strength, MPa
27	216	116	248	580
127	158	113	193	465
227	144	92	160	396
327	144	80	139	362
427	144	63	123	330
527	144	48	112	304
627	129	39	103	271
727	72	32	95	199
827	52	26	89	168
927	38	21	85	144
1027	28	17	80	125
1127	20	14	77	111
1227	15	11	74	100



2 *a* **calculated yield strength (YS) of MA956 and contributions from various components as functions of temperature and** *b* **calculated yield strength compared with results of neural network analysis**

density) to float off. The remaining particles coagulate and segregate making them ineffective as strengthening dispersoids (this is why yttria is not incorporated into the alloy by melting, but by mechanical alloying). At the same time, the other strengthening terms are unaffected by this procedure when the comparison is with a recrystallised MA956 specimen. Arc melting might seem a drastic method to eliminate dispersoid strengthening. It is nevertheless necessary since the yttria is added because of its stability in iron. Attempts to coarsen the dispersion would not only be ineffective but also unconvincing in the present context.

Figure 3 shows the optical microstructures and TEM images of the replicas of the specimens before melting and after melting. The optical micrographs show that the grain sizes of the two specimens are comparable whereas the replica images show that the experiment has been successful in removing fine dispersoids from the melted specimen. The number density of particles is clearly much larger in Fig. 3*c* than in Fig. 3*d*. This is reflected in the measured hardness values which were found to be in the range 248–253 HV10 (with mean 251 HV10) and 188–196 HV10 (with mean 192 HV10) for the unmelted and melted specimens respectively.

Nominal stress–nominal strain curves are presented in Fig. 4 which show that there is a reduction of 250 MPa in the yield strength for the melted specimen. This is in remarkable agreement with the calculated room temperature particle strengthening of 248 MPa, giving confidence in the model. (The incidental observation that a larger elongation is observed in the unmelted specimen is because the melted specimen fractured eventually by a cleavage mechanism. The reason for this has not been investigated in detail.)

Unrecrystallised MA956

There is a remarkable difference in the grain size of recrystallised MA956 ($\sim$10 mm) and the unrecrystallised alloy which has grains <1 μm in diameter. Grain size must therefore represent the major component of the reduction in strength following a recrystallisation heat treatment. If this is the case then the strength of the unrecrystallised MA956 is mainly that of the recrystallised specimen and the grain boundary strengthening. This assumption can be verified by comparing calculated grain size strengthening values with the measured difference between the strength of the recrystallised and unrecrystallised MA956 as manifested in the neural network analysis.[1] The Hall–Petch[20,21] relation is expressed as

$$\sigma_g = k_0 d^{-1/2} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (5)$$

where k_0 is a constant which is a measure of the grain boundary resistance and d is the grain size. According to equation (3), the value of k_0 for ferritic stainless steels is 15·9 MPa mm$^{-1/2}$ (Ref. 11).

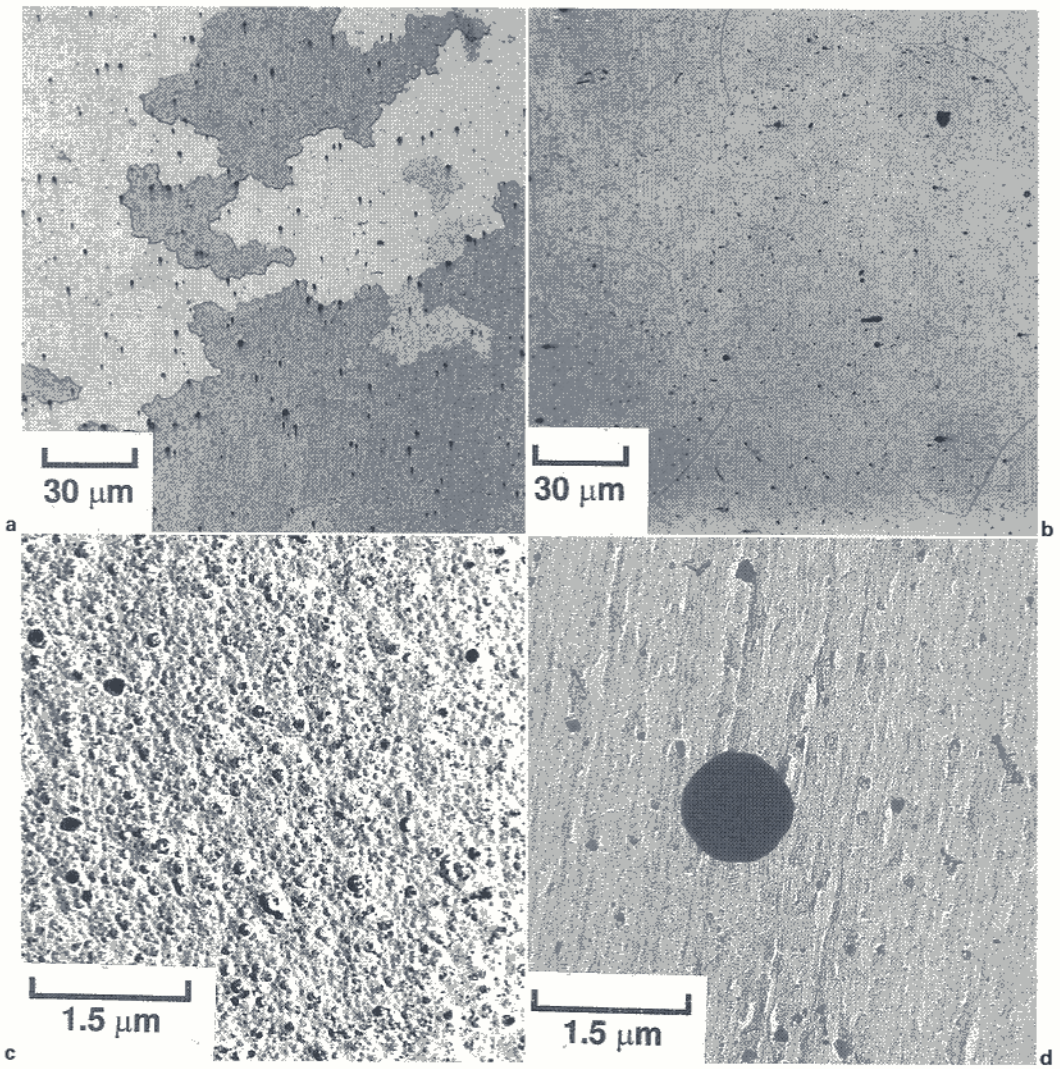
Although the grain size strengthening can be calculated using the Hall–Petch relation, there is no adequate theory for its temperature dependence. Therefore, the temperature dependence was estimated using the neural network model and it was assumed that the difference between the recrystallised and unrecrystallised specimens is essentially owing to the respective grain structures. The ratio of the difference relative to that at room temperature was used to scale the results according to equation (5). The contribution of dislocation strengthening was determined using the following expression[22]

$$\sigma_d = \alpha G b \rho^{1/2} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (6)$$

where $\alpha = 0{\cdot}05$ is the dislocation strengthening coefficient.[23] A relatively small value of $\sigma_d = 31{\cdot}4$ MPa was obtained and taken to be independent of temperature.

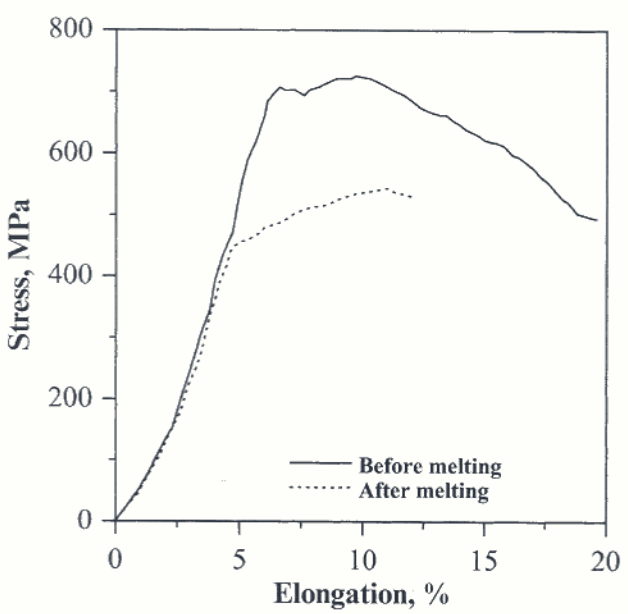
Figure 5 shows the calculated yield strength of the unrecrystallised MA956 as a function of the grain size in the range 0·5–1·0 μm. As before, the results are compared against the neural network interpretation of the experimental data. The yield strength can be explained rather well with the grain size set at about 1·0 μm. There is a slight increase in grain size strengthening with temperature over the range 30–300°C. This increase may not be significant given the uncertainty in the experimental data as apparent in the error bars, but it could be a consequence of strain aging associated with the small concentration of interstitial carbon in MA956. A similar effect is found in the temperature dependence of the strength in ordinary ferritic steels.[24]

The more significant decrease in the contribution from grain size strengthening occurs at higher temperatures. For austenitic steels, the reduction in grain size strengthening with increasing temperature has been associated with a weakening of dislocation locking effects at grain boundaries.[25] This might apply to MA956, but a more likely explanation is that there is dynamic recrystallisation accompanying deformation at high temperatures. This has been observed experimentally by Chou and Bhadeshia[26] during the hot deformation of MA956 and MA957 (another



3 Optical micrographs of MA956 specimens *a* before and *b* after melting showing similar coarse grained structure and TEM replica images *c* before and *d* after melting showing much larger number of particles in *c* than in *d*

MA-ODS ferritic strainless steel). The dynamic recrystallisation could be observed even when the deformation temperature was far less than the ordinary recrystallisation temperature of the alloys. This would also explain why the grain size contribution remains constant until ~500°C, as grain boundaries are not expected to be able to migrate until the iron and substitutional atoms acquire sufficient mobility.



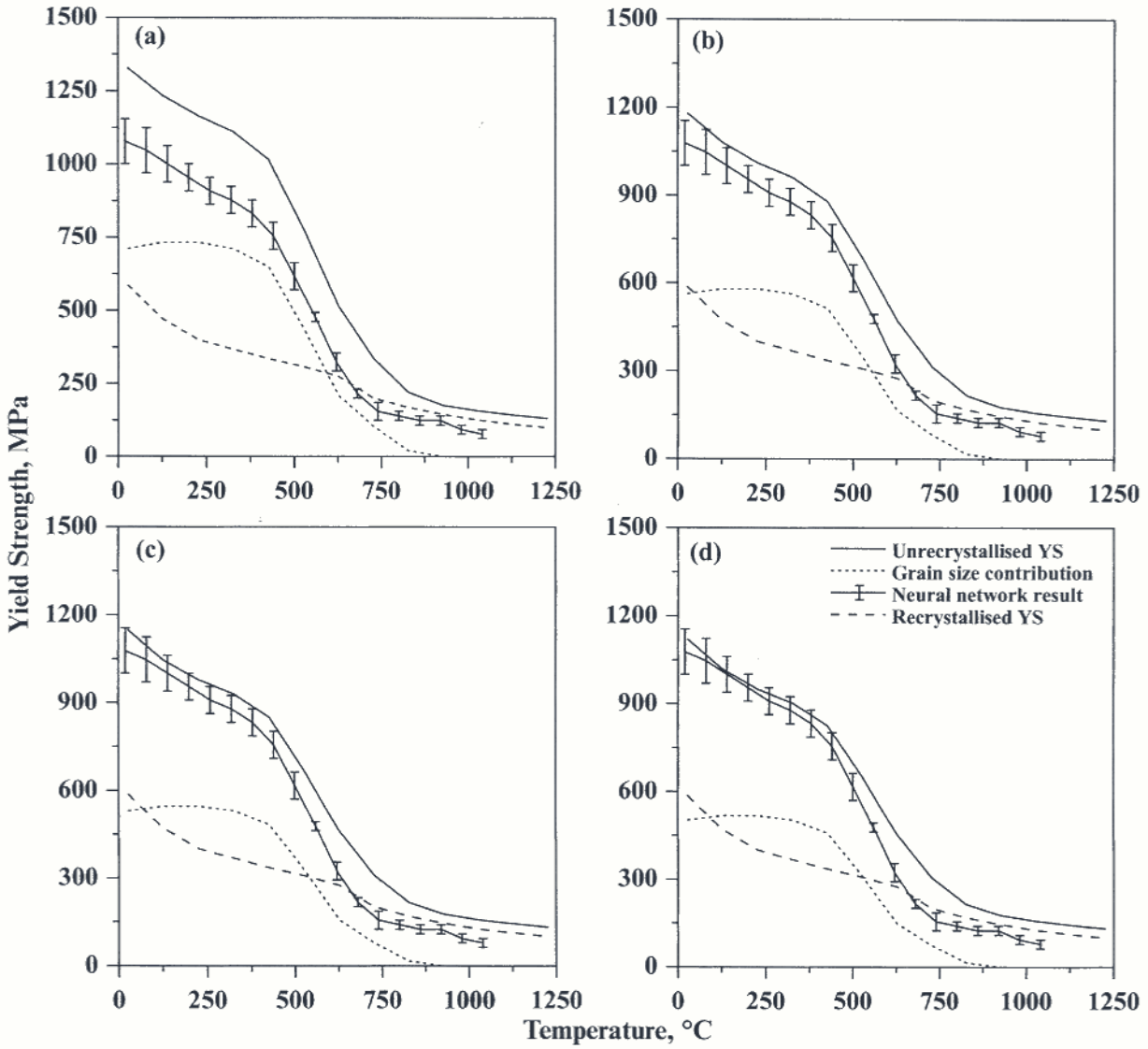
4 Tensile properties of MA965 before and after melting

To assess this, the grain sizes necessary to explain the reduction in the strengthening contribution σ_g were calculated from the difference between the recrystallised and unrecrystallised alloy strengths as a function of temperature (Fig. 6). The grain sizes estimated for all cases are seen to be quite reasonable in the sense that a fully recrystallised microstructure will have grains about 10–50 μm in size.[5]

Conclusions

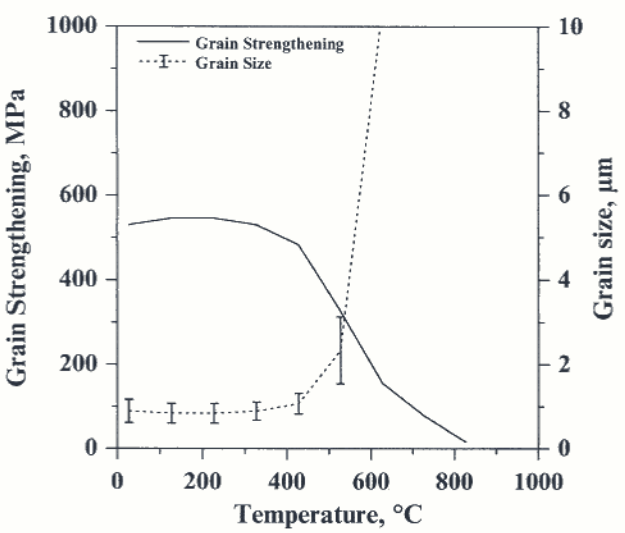
The ambient temperature yield strength of mechanically alloyed MA956 in the as processed condition originates from its ultra fine grain size, the intrinsic strength of ferritic iron, dispersoid strengthening via the yttria compounds, and finally, the dislocation density. The contributions of these components decrease in the order stated. The dispersoids contribute only ~250 MPa, but it is emphasised that their prime purpose is to provide creep resistance, a property not discussed here.

Recrystallisation has the effect of virtually eliminating grain size strengthening but leaving the other contributions essentially unchanged.



a 0·5 μm; *b* 0·8 μm; *c* 0·9 μm; *d* 1·0 μm

5 Calculated yield strength (YS) of unrecrystallised MA956 using given grain sizes and compared with neural network results: contribution from dislocation strengthening has been added to total unrecrystallised yield strength



6 Grain strengthening and estimated grain size of MA956 specimens which are unrecrystallised before testing as function of temperature

The temperature dependence of the strength has also been estimated. The relatively sharp decline in the strength in the recrystallised condition beyond ~500°C replicates the decrease in the strength of iron. It is believed that a further large temperature dependence in the case of the unrecrystallised specimen comes from dynamic recrystallisation during testing which reduces the grain size contribution to strength.

Finally, it is interesting that the authors' earlier paper[1] on neural network analysis of a vast quantity of published data was used to validate the physical models. Mechanically alloyed materials are notorious in their variability so this is a good method of providing an assessed experimental reference. Furthermore, the network provides estimates of uncertainty in the experimental data which are useful in the validation exercise.

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References

1. A. Y. BADMOS, H. K. D. H. BHADESHIA, and D. J. C. MACKAY: *Mater. Sci. Technol.*, 1998, **14**, 793–809.
2. E. A. LITTLE, D. J. MAZEY, and W. HANKS: *Scr. Metall. Mater.*, 1991, **25**, 1115–1118.
3. J. S. BENJAMIN: *Metall. Trans. A*, 1970, **A1**, 2943.
4. J. S. BENJAMIN and R. L. CAIRNS: in 'Modern development in powder metallurgy', (ed. H. H. Hausner), Vol. 5, 47–71; 1971, New York, Plenum.
5. A. ALAMO, H. REGLE, and J. L. BECHADE: *Novel Powder Process.*, 1992, **1**, 69.
6. H. CAMA and T. A. HUGHES: in 'Materials for advanced power engineering: Part II', 1497–1506; 1994, Dordrecht, Kluwer Academic Publishers.
7. H. REGLE: PhD thesis, 'Alliages ferritiques 14/20% de chrome renforces par dispersion d'oxydes', Universite de Paris Sud, 1994.
8. N. HANSEN and H. LILHOLT: in 'Modern development in powder metallurgy', (ed. H. H. Hausner), Vol. 5, 339–365; 1971, New York, Plenum.
9. C. H. YOUNG and H. K. D. H. BHADESHIA: *Mater. Sci. Technol.*, 1994, **10**, 209–214.
10. W. C. LESLIE: *Metall. Trans.*, 1972, **3**, 5–26.
11. D. B. LEWIS and F. B. PICKERING: *Met. Technol.*, 1983, **10**, 264.
12. R. S. W. SHEWFELT and L. M. BROWN: *Philos. Mag.*, 1977, **35**, 945–962.
13. R. S. W. SHEWFELT and L. M. BROWN: *Philos. Mag.*, 1974, **30**, 1135–1145.
14. A. KELLY and R. B. NICHOLSON: 'Progress in materials science'; 1963, New York, Macmillan.
15. H. S. UBHI, T. A. HUGHES, and J. NUTTING: in Proc. Conf. 'Frontiers of high temperature materials I', (ed. J. S. Benjamin), 33; 1981, New York, Inco Alloys.
16. R. W. K. HONEYCOMBE and H. K. D. H. BHADESHIA: 'Steels: microstructure and properties', 2nd edn; 1995, London, Edward Arnold.
17. A. SEEGER: *Philos. Mag.*, 1954, **45**, 771–773.
18. P. KRAUTWASSER, A. CZYRSKA-FILEMONOWICH, M. WIDERA, and F. CARSUGHI: *Mater. Sci. Eng.*, 1994, **A117**, 199–208.
19. P. DUBIEL, W. OSUCH, M. WRÓBEL, A. CZYRSKA-FILEMONOWICH, and P. J. ENNIS: in 'Materials for advanced power engineering: Part II', (ed. D. Coutsouradis *et al.*), 1523–1532; 1994, Dordrecht, Kluwer.
20. E. O. HALL: *Proc. Phys. Soc. (London)*, 1951, **64**, 747.
21. N. J. PETCH: *J. Iron Steel Inst.*, 1953, **174**, 25.
22. G. E. DIETER: 'Mechanical metallurgy', 3rd edn, 232; 1986, New York, McGraw-Hill.
23. D. HULL and D. J. BACON: 'Introduction to dislocations', 3rd edn, International Series on Materials Science and Technology, 246; 1984, Oxford, Pergamon.
24. H. J. TAPSELL: 'Creep of metals'; 1931, Oxford, Oxford University Press.
25. V. K. RAO, D. M. R. TAPLIN, and P. R. RAO: *Metall. Trans. A*, 1975, **6**, 77–85.
26. T. S. CHOU and H. K. D. H. BHADESHIA: *Mater. Sci. Technol.*, 1995, **11**, 1129–1138.