Through Thickness Microstructural Gradients in 7475 and 2022 Creep - Ageformed Bend Coupons

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Abstract. In creep-ageforming a material experiences continuously variable bending stresses through its thickness, from tensile to compressive, which are maximum at the surfaces. This can potentially result in through thickness microstructural gradients, due to interactions between the bending stresses, creep, and precipitation occurring during ageing, that can alter a component's performance. The aim of the work reported here was to develop an understanding of these effects in an industrial creep ageforming process. For this purpose two aerospace alloys, 7475 and 2022, were ageformed by Airbus UK using industrial scale ageforming tools. Their microstructures were analysed in detail by TEM, and SAXS, which revealed significant through thickness microstructural changes after forming.

Introduction

To date, creep-ageforming has been primarily used in the civil aerospace industry to form upper wing skins. Although investigations are being carried out to expand the application of ageforming to integral and damage tolerant parts, where there is a requirement for the implementation of new alloy – temper solutions [1], the types of microstructural interactions that might be expected in the ageforming process have currently only been investigated on a laboratory scale, in constant stress-ageing tests. These studies have shown that the applied stress may affect the precipitation behaviour during ageing in some alloys [2-7]. Observations include; preferential habit plane variant selection and changes in the precipitate morphology and size of, principally, plate shaped phases [5-8]. Most of this research has been restricted to Al-Cu based alloys [2,3,6]. However, a detailed microstructural investigation has never been reported for an industrial ageformed component. In the industrial case the formed coupon is subjected to bending stresses, depending on the part thickness and curvature of the forming tool, which are maximum and of opposing sign at the top and bottom surfaces and continuously vary through the thickness, being zero at the neutral axis. Furthermore, these stresses are not constant and progressively relax during the forming cycle. Depending on the alloy system, this could affect the strengthening phases that develop on ageing differently at different positions through the plate thickness. The dislocation activity associated with the creep relaxation required to obtain the component shape might also be expected to affect the ageing behaviour of the material [9-10]. These interactions can potentially result in through thickness microstructural gradients that could alter a components performance, post-forming.

The objective of the present study was to establish how significant any material interactions might be in influencing the through thickness microstructure in an industrial ageformed component. To this end, detailed TEM and SAXS analysis has been performed on industrially ageformed bend coupons produced in two commercial alloys representative of the 7xxx and 2xxx alloy classes; namely 7475 and 2022.

Experimental Procedure

20 mm thick plates from two 7475 and 2022 alloys were ageformed, following standard industrial practice, in a 3180 mm radius single curvature tool, using an autoclave and vacuum bagging system, at Airbus UK's Broughton facility. The 2022 material was supplied in a solution treated and stretched T3 temper, and the 7475 alloy in a pre-aged TAF temper, which included the first lower temperature artificial ageing step of a damage tolerant T73 two stage-ageing treatment. The alloys were artificially aged in the forming tool to T851, for 2022, and to complete the last higher temperature stage of the T7351 temper for 7475. The bend coupons were formed along the rolling direction. The maximum surface strains were ± 0.0026 at the outer and the inner surface, which corresponds to a maximum tensile and compressive principal stress of ± 185 MPa, prior to any relaxation. For a baseline comparison, stress free samples of the same materials were heat treated in a laboratory furnace programmed to replicate the industrial thermal practice. Hardness profiles were measured through the plate thickness on the ageformed and control samples. Specimens were extracted from the plates top surfaces, quarter, mid plane, and three quarter positions, and bottom surfaces for qualitative microstructural characterisation by TEM and, for 7475, in order to measure the size of the second phase particles by SAXS using the x-ray synchrotron source at the CCLRC, Daresbury, UK. After instrument and background corrections the SAXS data was analysed following the method described in [11] to obtain the Guinier radius (R_g), by using an iterative procedure involving fitting the slope of integrated intensity vs scattering vector (I vs q^2) plots in the region of the $0.8 < qR_g < 1.8$ Kratky plot. By assuming random alignment of the precipitates, this was converted into a 'mean sphere radius' using the following formula; $R_o = \sqrt{\frac{5}{3}} R_g$ [12].

Results

Through Thickness Behaviour of the 2022 T851 Ageformed Coupon. After ageing to the T851 temper the dominant phase observed in the 2022 alloy was θ' , with some remaining θ'' , some Ω , and a very low volume fraction of S and σ . In Fig. 1 TEM images are shown for the through thickness positions of the ageformed plate, along with inserts showing small sections of $\langle 001 \rangle$ zone axis selected area diffraction patterns (SAD), from around the $\{110\}_{Al}$ position, to highlight the relative intensities of the reflections from the habit plane variants of θ' (streaks) and Ω (spots). From the images and diffraction data, very strong preferential alignment of the θ' phase can be seen parallel to the tensile, or perpendicular to, the compressive principal stress axis found at the plate outer and inner surfaces. The degree of preferred orientation reduces for the $\frac{1}{4}$ and $\frac{3}{4}$ positions, as might be expected because the initial bending stress is inversely proportional to the distance from the neutral axis. However, a random distribution over the available habit plane variants is not observed at the mid plane. In contrast, the θ' plates are still aligned in a sense indicative of the presence of a weak compressive stress parallel to the rolling direction, which suggests the neutral axis has shifted towards the outer surface of the plate.

From close inspection of the enlarged regions of the diffraction patterns in Fig. 5, it can be seen that there are four spots present on diagonals between the θ' streaks, originating from the four $\{111\}_{Al}$ habit plane variants of the Ω phase. These spots are always present with approximately symmetric intensities, irrespective of the sample depth position and stress level, which demonstrates that there is no preferential alignment of the Ω phase in the ageformed coupon.

Through thickness hardness distributions for the 2022 alloy, measured on the formed coupon and the stress-free aged control sample, are shown in Fig. 2. In both curves there is a small dip in hardness at the plate centre caused by macrosegregation in the original DC cast billets, but there is not a great deal of difference between the two sets of data. The results for the stress-free aged sample are all systematically shifted to a slightly higher value, which is probably due to a small difference in the ageing temperature between the industrial autoclave and laboratory furnaces, and do not show a strong influence of the variation in precipitate alignment through the plate thickness.

Fig.1 TEM images for the 2022 alloy from the through thickness positions of the age formed plate, along with inserts showing small sections of $\langle 001 \rangle$ SAD patterns to highlight the relative intensities of the θ' and Ω reflections; (a) the outer surface (tensile) (b) the $\frac{1}{4}$, (c) the mid plane, (d) the $\frac{3}{4}$ position and (e) the inner (compressive) surface. The arrows indicate the direction of the principal stress axis/rolling direction relative to each micrograph.

Fig. 2 Through thickness hardness profiles measured from the 2022 and 7475 alloy coupons after ageforming, compared to the stress-free, identically aged, control samples.

Through Thickness Behaviour of the 7475 T7351 Ageformed Coupon. In the T7351 over-aged temper the 7475 alloy's microstructure typically contains a mixture of dislocation nucleated η in a matrix of η' [7]. In Fig. 3 TEM images are given for the 7475 alloy from the through thickness positions of the age formed plate, along with inserts showing small sections from $\langle 111 \rangle$ zone axis SAD patterns, to highlight the relative intensities of the η' and η reflections. Through thickness average particle sizes determined from SAXS analysis and hardness profiles are given in Figs. 4 and 2. Although preferential nucleation on habit plane variants sympathetic with the applied stress axis has been reported in 2xxx alloys [2-7], in previous studies of 7475 aged under a constant stress no preferential alignment of the η' or η phases was observed [7] in this temper condition. Here, again, no preferential alignment of either of these phases could be seen at any depth position, by TEM.

The SAXS patterns also showed a broadly isotropic intensity distribution in the ND-TD plane (Fig. 4a).

Fig. 3 TEM images for the 7475 alloy from the through thickness positions of the age formed plate, along with inserts showing small sections of $\langle 111 \rangle$ SAD patterns to highlight the relative intensities of the η' and η reflections; (a) the outer surface (tensile) (b) the $1/4$, (c) the mid plane, (d) the $3/4$ position and (e) the inner (compressive) surface. The arrows indicate the direction of the principal stress axis/rolling direction relative to each micrograph.

Fig. 4 SAXS diffraction data for the 7475 bend coupon after ageforming; in (a) the SAXS patterns (1 -outer tensile surface, 2- $1/4$ depth, 3 -mid plane, 4 - $3/4$ depth position and 5 -inner compressive surface), (b) the mean particle radius as a function of depth, and (c) integrated intensity vs. scattering vector profiles from the two surfaces and mid plane, using a 4 m camera to capture scattering vector intensities in the range of the larger η particles.

Although in this temper no precipitate alignment was detected, coarsening was observed to occur in the regions of the ageformed coupon that had experienced the largest bending stresses. In Fig. 3 the average precipitate size can be seen to be greater, and number density lower, at the plate

surfaces. The precipitate size reduces at the $\frac{1}{4}$ and $\frac{3}{4}$ positions to reach a minimum at $\sim$ the plate mid plane. However, this effect was not symmetric about the plate centre and it can be clearly seen in Fig. 2e that the precipitate size was coarsest at the compressively loaded inner surface of the plate. Analysis of selected area diffraction patterns (Fig. 3) revealed that the relative intensities of the η' and η reflections also changed with depth position, indicating that the material contained a higher fraction of η , compared to η' , at the plate surfaces. In particular, at the plate inner surface, where the average precipitate size was greatest, the diffraction intensities were dominated by η_1 , η_2 , and η_4 , variants of the η phase and very little η' was seen.

The SAXS technique provides more statistically reliable volume averaged data than the TEM and in Fig. 3 the mean spherical radius measured using this method is plotted as a function of depth through the formed plate. This data confirms the TEM observations, in that there is an asymmetric increase in average precipitate size towards the plate surfaces, which is greatest at the compressively loaded inner surface. The particle size measured at the plate centre was virtually identical in the stress free control samples. Through thickness hardness distributions measured on the formed coupon and stress-free aged control sample are shown in Fig. 2, and on the whole agree with the trends seen in the TEM and SAXS results. Overall the hardness values for the ageformed plate are lower and are more reduced, relative to the control, at the compressive inner surface of the billet.

Discussion

Taken together the TEM, SAXS, and hardness data show that there is a subtle, but significant, effect of the ageforming process on the precipitation behavior of the 2022 and 7475 alloys, which results in a through thickness microstructural gradient in a formed component. In terms of the direct effect of an externally applied uniaxial stress, this has been shown to have the greatest influence on nucleation kinetics, where it can reduce the energy barrier for a thin plate shaped nucleus with a large normal misfit strain, as is typically seen for GPZs, θ'' and the θ' and Ω precipitates in the 2xxx series alloy [3-7]. This naturally results in preferred alignment of such phases on habit plane variants orientated so that the nucleus misfit can take maximum advantage of the superimposed strain field. In comparison, the direct effect of the applied stress on precipitate growth is thought to be minimal [3-7]. Preferred alignment was not seen in the 7475 alloy, which suggests that there is little direct influence of the bending stress on the activation energies for nucleation of η' and η in this alloy. The behaviour in the 2xxx series alloy is as might be expected from constant load studies [4,6,7], where for θ' the degree of plate alignment increases with stress level and favours two $\{100\}_{Al}$ habit plane variants parallel to the tensile, and one habit plane variant normal to a compressive, principal stress axis. Although in constant stress tests Ω is also found to be aligned by the presence of an applied stress, this was not observed in the industrial bend coupon and results from the higher threshold stress for alignment of Ω compared to θ' ($\Omega \sim 110$; $\theta' \sim 30$ MPa [7]). Ω 's threshold stress is initially lower than the maximum surface bending stresses, but the stresses rapidly relax to below this critical level during the first few hours of ageforming and before Ω nucleates, as it appears later in the ageing sequence.

In the 7475 alloy, although there is no direct effect of the bending stresses on nucleation, significant particle coarsening occurs in the most highly stressed regions. This results primarily from more rapid over-ageing, when heat treated to the T73 temper, which causes an increase in the average precipitate size and volume fraction of the η phase at the expense of η' . This phenomenon can be attributed to the initial bending stress acting as a driving force for the dislocation glide and climb activity, which occurs in the power law creep regime that gives rise to the creep relaxation in the ageforming cycle. Similarly, stretching prior to aging is known to accelerate over-ageing in 7xxx alloys, by providing dislocation nucleation sites for the equilibrium η phase [13]. In comparison in ageforming the plastic creep strain is relatively small (<0.0015) and therefore the

dynamic nature of the precipitation and simultaneous creep processes must have a disproportionate effect on the ageing kinetics.

The microstructural gradients in both alloys show that the bending stresses are not relaxed symmetrically in industrial forming. This probably results from the influence of the stretching operation which gives rise to a lower compressive yield stress and a residue 'Bauschinger effect' allowing faster relaxation in compression than tension. Another possible source of asymmetry could be caused by stresses generated due to friction between the die and plate in conjunction with differential thermal expansion of the parts and volume changes to the material, associated with solute rejection from solid solution during ageing [14].

Summary

In creep-ageforming a material experiences continuously variable bending stresses through its thickness, from tensile to compressive, which are maximum at the surfaces. In the two materials studied this has been shown to have a significant effect on the through thickness microstructure that develops in an ageformed component. In 2xxx alloys, which contain thin plate precipitates with a large normal misfit strain, this causes precipitate alignment on habit plane variants that minimise the strain energy barrier to nucleation, which increases towards the plate surfaces. In 7xxx alloys there is an indirect effect of the dislocation activity occurring during creep relaxation which results in more rapid overageing near the plate surfaces. The creep relaxation behaviour itself appears to occur asymmetrically, with greater relaxation occurring on the compressive side of the plate.

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References

- [1] M.C. Holman: J.of Mechanical Working Technology, Vol. 20, (1989), p. 477.
- [2] M. W.F.Hosford, and S.P.Agrawal: Metall.Trans.A, Vol. 6A, (1975), p. 87.
- [3] T.Eto, A.Sato and T.Mori: Acta. Met. Vol. 26, (1978), p. 499.
- [4] H.Hargarter, M.T.Lyttle, E.A. Starke: Mater. Sci. and Eng., Vol. A257 (1998), p. 87.
- [5] B.Skrotzki, G.J.Shiflet, E.A.Starke Jr: Met. and Mat. Tra. A, Vol. 27A, (1996), p. 3431.
- [6] A.W.Zhu, E.A Starke Jr: Act. Mat., Vol 49, (2001), p. 2285.
- [7] D.Bakavos, P.B.Prangnell, R.Dif: Mater. Sci. Forum, Vol . 28, (2004), p. 124.
- [8] D.Bakavos, P.B.Prangnell, B.Bes.Eberl, J.G Grossman; in the present proceedings.
- [9] R. N. Lumley, A. J. Morton and I. J. Polmear: Acta Mater., Vol. 50, (2002), p. 3597.
- [10] R.F.Robey, P.B.Prangnell, R.Dif: Mater. Sci. Forum, Vol . 28, (2004), p. 132.
- [11] A. Deschamps: *Analytical Techniques for aluminium alloys, Handbook of Aluminium, Vol. 2*, p.155 (Marcel Dekker, Inc., New York, USA, 2003, ed.G.E. Totten and D.S.MacKenzie)
- [12] M.R.Baig, S.Gupta, S.Messoloras and R.J.Stewart: J.Appl.Cryst. Vol. 24, (1991), p. 349.
- [13] A. Deschamps, F.Livet and Y.Brechet: Acta. Mater., Vol. 47, (1999), p. 281.
- [14] H.J.Axon and W.Hume-Rothery: Proc.Roy.Soc, Vol. A193,(1948), p. 1.