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Effect of extrusion parameters on the microstructure and properties of an Al–Li–Mg–Zr alloy

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The effect of the extrusion temperature and the extrusion ratio on the microstructure and mechanical properties of an Al–Li–Mg–Zr alloy in the as-extruded, solution-treated and aged conditions were investigated. It was found that an increase in the extrusion temperature and ratio increased the degree of recrystallization in the extrudate. An increase in the extrusion temperature also increased the sub-grain size, whereas the extrusion ratio did not affect the sub-grain size. The strength of the extrudates decreased and the ductility increased in the as-extruded condition with increases in the extrusion temperature and the extrusion ratio. However, in the solution-treated condition, although the effect of the extrusion ratio on the mechanical properties was found to be the same as in the as-extruded condition, the trend with the extrusion temperature changes and the extrusion temperature was not simple in its influence on the mechanical properties. The fracture toughness remained almost unaffected by variation either in the extrusion temperature or the extrusion ratio.

1. Introduction

Al–Li alloys are potentially important for the aerospace industry as the addition of 1%* Li reduced the density by 3% and increased the elastic modulus by 6% [1]. Direct extrusion is an important forming process, and in a typical modern transport aircraft the use of this product form may be as high as 28% [2]. In Al–Li alloys containing Zr, the material properties are dependent upon the process parameters, even after solution-soaking and ageing treatments, as the hot-worked structure is partially retained after these treatments. Microstructural features which influence the mechanical properties and which are determined by the extrusion conditions include the sub-grain structure the degree of recrystallization. However, only a few studies [3–9] have been made of the effect of the extrusion parameters on the microstructure and mechanical properties of Al–Li alloys. In this paper the effect of the extrusion temperature and ratio on the recrystallization and recovery behaviour and their influence on the mechanical properties in the as-extruded (AE), solution-treated (ST), under-aged (UA), peak-aged (PA) and over-aged (OA) conditions of an Al–Li-based alloy are systematically investigated.

2. Experimental procedure

The material for the present investigation was received from ALCOA, USA in the form of ingots 114 mm in diameter and 406 mm long, cast by a permanent mould technique, having a nominal composition Al–2.5Li–2Mg–0.08Zr. The material was homogenized at 535 °C for 24 h. The homogenized ingots were machined down to 72 mm in diameter and 90 mm in length for extrusion. Extrusion was carried out in an ENEFCO vertically acting 5 MN press with a fast-ram-approach facility. The billets were heated to the extrusion temperature in a Banyard Metalheat induction furnace adjacent to the press, and billets were then transferred pneumatically into the container of the extrusion press. The heating rate up to the extrusion temperature was 125 °C min^{-1}, and on reaching the extrusion temperature the billet was given a 2 min soak. The container was maintained at 25 °C below the extrusion temperature to avoid significant heat loss from the billet during extrusion. Extrusion was carried out at three different temperatures, namely 350, 400 and 525 °C, at a 30:1 ratio, and at two different extrusion ratios, namely, 30:1 and 20:1, at the 400 °C temperature. The ram speed was maintained at 3 mm s^{-1} for all the extrusions. The extrusions were produced in a rod shape, and were press quenched in order to retain the hot-worked structure.

A standard solution treatment of 530 °C for 40 min was given to all the extrudates. The solutionized extrudates were subsequently quenched in water at room temperature. The solutionized and quenched extrudates were aged in fan-driven air-circulating ovens at 190 °C.

For the observation of the recrystallization behaviour, longitudinal-section specimens were examined.

* Weight percentage everywhere.

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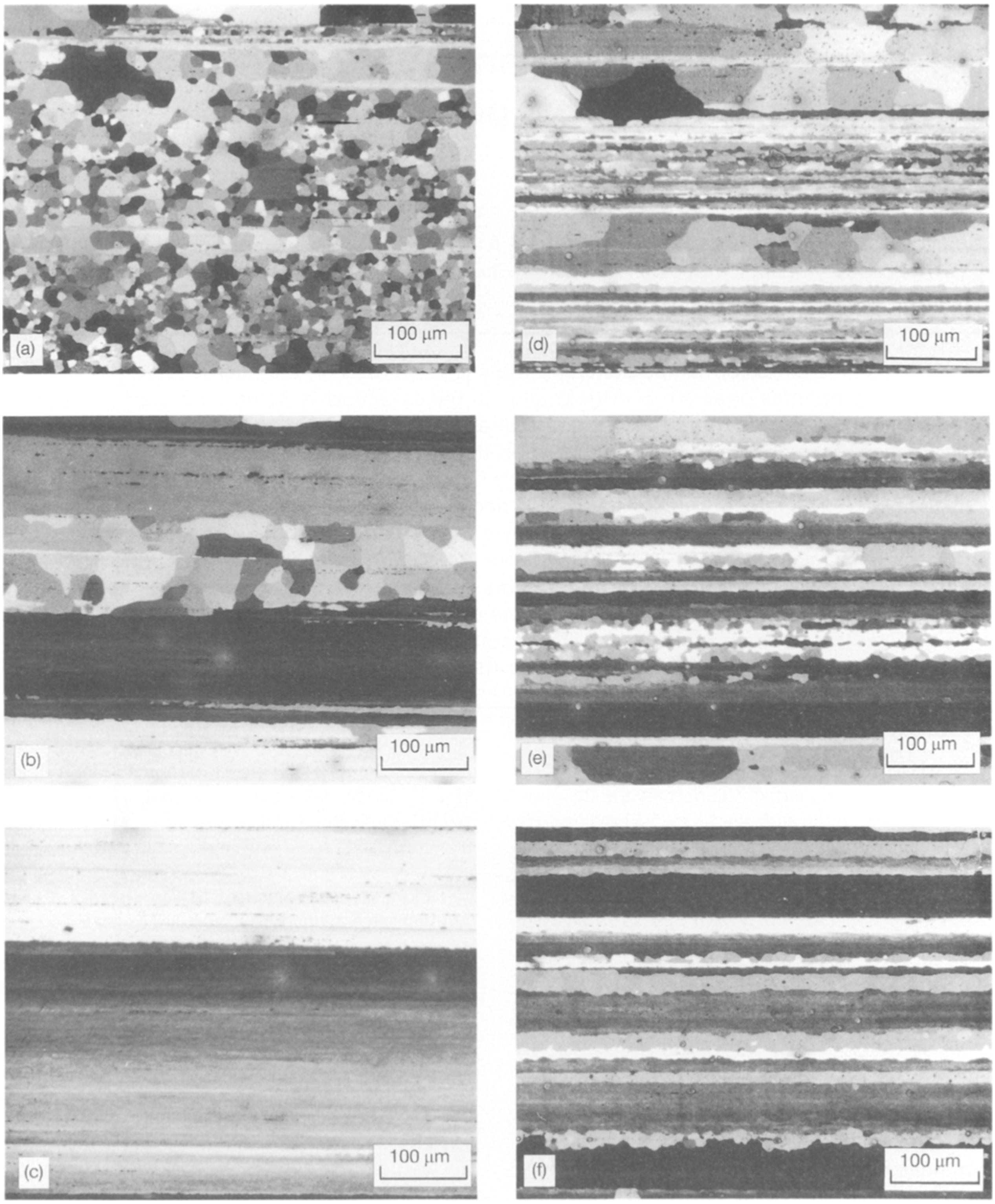

Figure 1 Optical micrographs of the longitudinal section of the as-extruded alloy, extruded at 350 °C, in a 30:1 ratio, at: (a) the surface showing the recrystallized microstructure, (b) the mid-radius showing a layer of recrystallized grains amidst the unrecrystallized grains, and (c) the core showing the unrecrystallized microstructure. Optical micrographs of the longitudinal section of the as-extruded alloy, extruded at 525 °C, in a 30:1 ratio, at: (d) the surface showing a recrystallized microstructure, (e) the mid-radius showing layers of recrystallized grains amidst the unrecrystallized grains, and (f) the core showing an unrecrystallized microstructure with the nucleation of few recrystallized grains, giving a scalloped appearance.

These specimens were ground using 600- and 1200-grade silicon-carbide papers, polished on 14, 6 and 1 µm diamond pastes and anodized in Barker's reagent at room temperature at 20 V. The anodized specimens were examined in polarized light under a Nikon optical microscope. The sub-grain structure was examined by transmission electron microscopy (TEM); the TEM method was outlined in a previous paper [10].

Standard Hounsfield-number-12 specimens were prepared and tested on an Instron universal testing machine at room temperature at a strain rate of 5.2

$\times 10^4$ s^{-1}. The ultimate tensile strength (UTS), 0.2% proof strength (0.2%PS) and percentage elongation (%EL) were measured from tensile-load versus elongation curves. Toughness measurements were made using short-rod-fracture-toughness specimens specified by Terra Tek and recommended by ALCOA [11]. This method produces excellent correlation with K_{Ic}-values determined in accordance with ASTM method E999. The fracture-toughness specimens were prepared from longitudinal sections of the extrudates, and the load was applied at 90° to the extrusion direction.

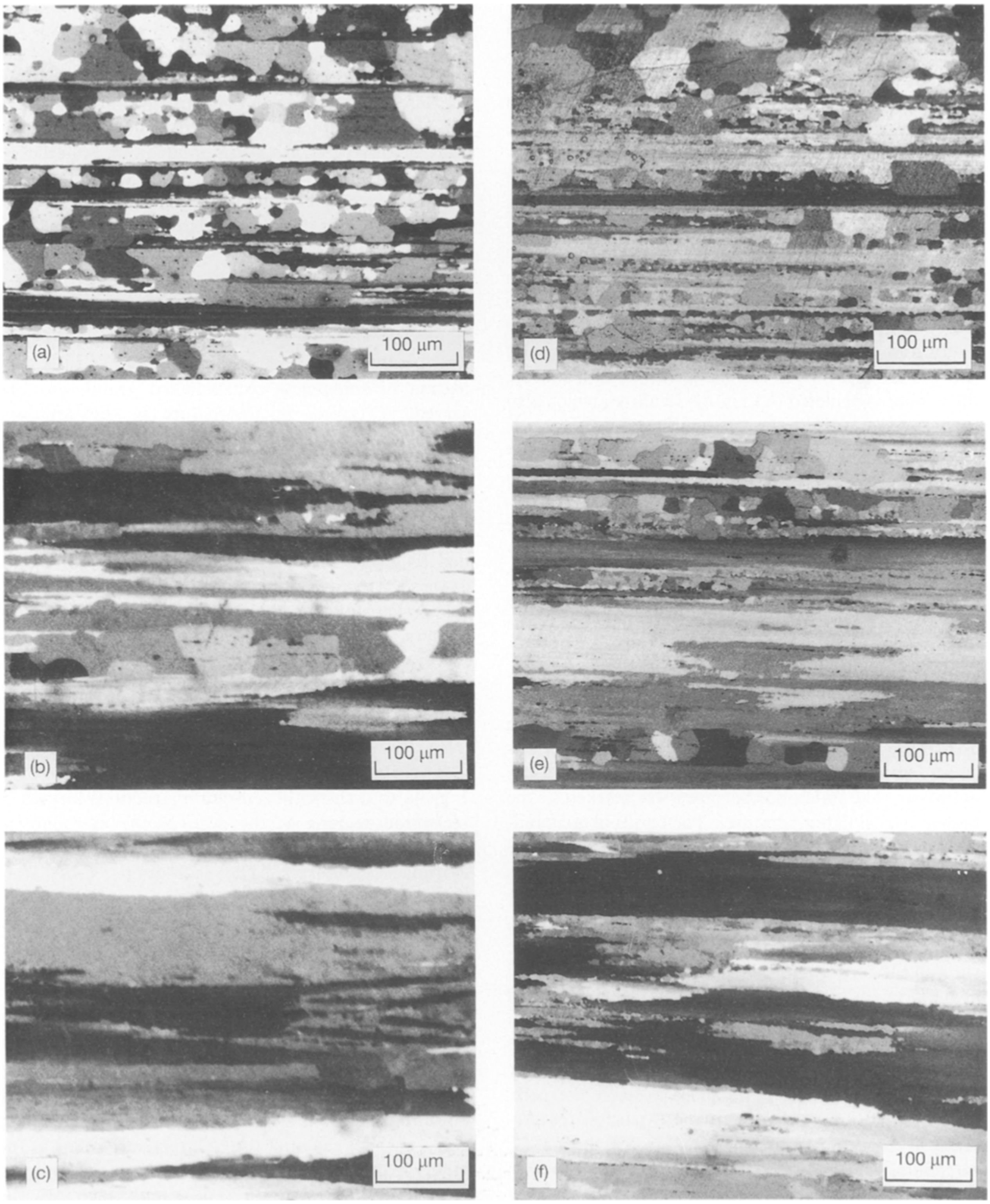

Figure 2 Optical micrographs of the longitudinal section of the as-extruded alloy, extruded at 400 °C, in a 30:1 ratio, at: (a) the surface showing a recrystallized microstructure, (b) the mid-radius showing a layer of recrystallized grains amidst the unrecrystallized grains, and (c) the core showing an unrecrystallized microstructure. Optical micrographs of the longitudinal section of the as-extruded alloy, extruded at 400 °C, in a 20:1 ratio, at: (d) the surface showing a partially recrystallized microstructure, (e) the mid-radius showing layers of recrystallized and unrecrystallized grains, and (f) the core showing an unrecrystallized microstructure.

3. Results and discussion

3.1. Effect of the extrusion temperature and ratio on the as-extruded microstructure

Figs 1 and 2 show the as-extruded microstructure at the surface, mid-radius and core of the extrudates along the longitudinal direction of the extrudates at various extrusion temperatures and ratios. During extrusion, the grains are elongated parallel to the extrusion direction. Therefore, the longitudinal microstructure appears in the form of parallel bands. At all the extrusion temperatures, the surface was fully recrystallized, the layer adjacent to the surface was partially recrystallized, a few bands of recrystallized grains were observed up to very near the core, and the core was largely unrecrystallized. Al has a high stacking-fault energy, implying that partial dislocations will combine to form full dislocations which promotes cross slip and hence recovery. Thus, the recovery process takes place easily in Al alloys, reducing the driving force for recrystallization, and the microstructure remains by and large unrecrystallized in these alloys. However, at the surface of the extrudate, the strain and the temperature are relatively larger, and this tends to promote recrystallization. In addition, the surface is depleted in Li in Al–Li alloys, which also favours recrystallization at the surface [12].

At the surface, the recrystallized grains were finer at 350 °C, and, as the extrusion temperature increased to 400 and 525 °C, these grains tended to become larger, as can be seen in Figs 1a and d and 2a. At lower extrusion temperatures, the nucleation sites for recrystallized grains are more numerous due to the greater stored energy, whilst the kinetics of the growth of the recrystallized grains is slow, giving rise to the finer size of the recrystallized grains. In the case of the extrudates extruded at 350 °C, the recrystallized grains formed in the interior are larger (Fig. 1b) than those formed at the surface of the extrudate. This is again due to the fewer nucleation sites, owing to the lower strain in the interior compared to the surface of the extrudate. The difference becomes less marked at the higher extrusion temperatures. The bands of recrystallized grains in the interior of the extrudates were encountered more frequently in the case of extrudates extruded at higher temperatures, indicating a greater amount of recrystallization at higher temperatures, and in the case of the 525 °C extrudate, even the core showed the formation of some recrystallization nuclei at the original grain boundaries, giving it a scalloped appearance (Fig. 1f). The increase in the deformation temperature decreased the critical strain necessary to initiate recrystallization, and this is the reason why some recrystallization takes place even at the core, where the strain is smaller, as the extrusion temperature is increased. A peculiar feature of the 525 °C extrudates was the presence of some fine bands within the original grains and of very fine recrystallized grains inside those bands. In some alloys, deformation bands are formed within the original grains during hot working, and recrystallized grains are formed within those bands giving such an appearance. However, there does not seem to be any reason why this process should take place at 525 °C and not at the lower extrusion temperatures. In any case, in the absence of any further evidence, no explanation is offered for this particular feature.

Decreasing the extrusion ratio reduces the amount of recrystallization, as can be seen in Fig. 2. In the case of the extrudates extruded at a 20:1 ratio, even the surface was not fully recrystallized (Fig. 2d), and in the interior of the extrudate only clusters of recrystallized grains were observed in a few places (Fig. 2e) and not the complete bands of recrystallized grains as was the case for the extrudates extruded at a 30:1 ratio at the same temperature and ram speed (Fig. 2b). The reason for this is that increasing the extrusion ratio increases the strain, which increases the amount of inhomogeneous deformation at the original grain boundaries, thus increasing the driving force for the formation of the recrystallization nuclei, giving rise to more recrystallization in the as-extruded microstructure.

3.2. Microstructural changes accompanying the solution treatment

In general, with the solution treatment, the growth of the already existing recrystallized grains and the nucleation of some new recrystallized grains was observed. A typical microstructure in the solution-treated condition is shown in Fig. 3 for the extrudates extruded at 400 °C, a 30:1 ratio and a 3 mm s^{-1} ram speed prior to the solution treatment and the water quenching. The already recrystallized grains formed during extrusion at the surface of the extrudate continued to grow. Some grains grew much larger at the expense of the others, as shown in Fig. 3a. There were more recrystallized grains in the interior of the extrudate and the core, which, though still largely unrecrystallized, showed the formation of a few recrystallization nuclei at the original grain boundaries in the solution-treated condition (Fig. 3c).

3.3. Sub-grain sizes

Figs 4a to d show the sub-grain structures formed in recovered regions at the core of the extrudate at various extrusion temperatures and ratios in the as-extruded condition. The sub-grain sizes were ~ 2.8 μm at 350 °C, ~ 3.5 μm at 400 °C and ~ 6.8 μm at an extrusion temperature of 525 °C and a 30:1 ratio. The extrudates extruded at 400 °C and a 20:1 ratio had sub-grains of ~ 3.5 μm in size. So, the sub-grain size increased with increasing extrusion temperature, and it remained unaffected by the variation in the extrusion ratio investigated. An increase in the extrusion temperature decreased the generation rate and increased the annihilation rate of the dislocations, resulting in a lower equilibrium dislocation density which gave rise to the larger sub-grains at the higher extrusion temperatures. On the other hand, an increase in the extrusion ratio simply increases the total amount of the strain, which does not have any effect on the sub-grain size.

With the solution treatment, the sub-grain sizes in the recovered region were found to be the same (Fig. 4e). However, a few large sub-grains were also

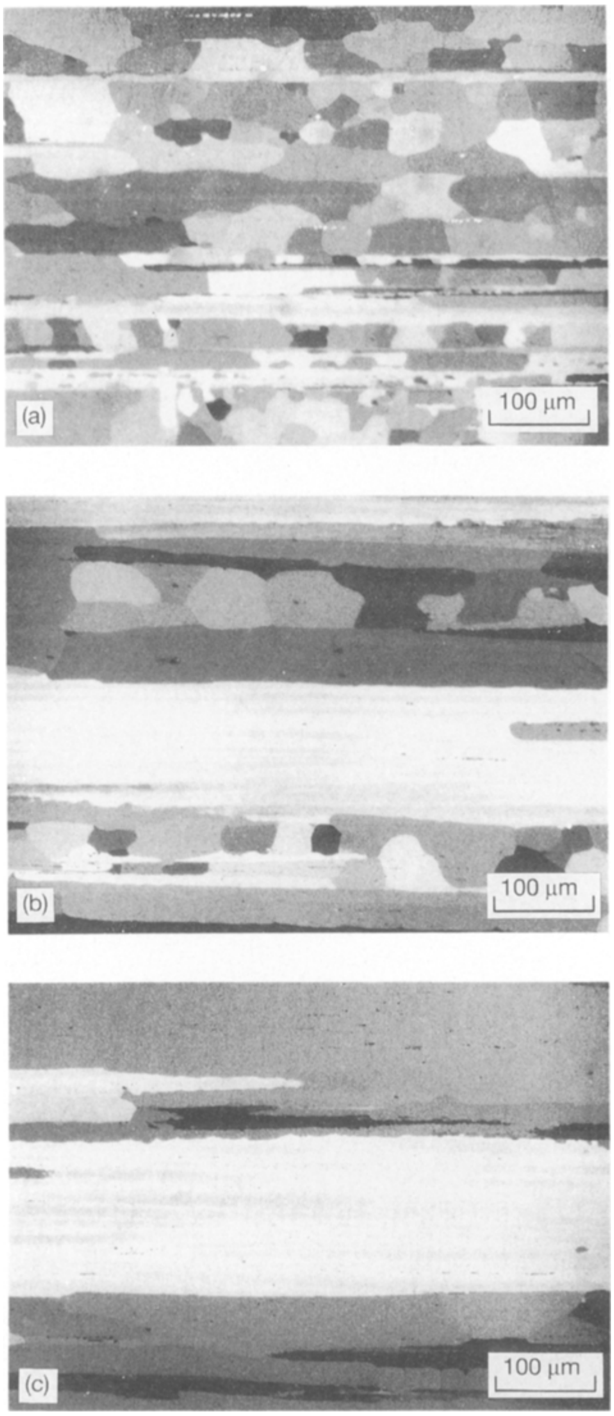

Figure 3 Optical micrographs of the longitudinal section of the solution-treated alloy at: (a) the surface showing a recrystallized microstructure, (b) the mid-radius showing layers of recrystallized grains amidst the unrecrystallized grains, and (c) the core showing an unrecrystallized microstructure with the nucleation of few recrystallized grains.

observed in association with the smaller sub-grains. One such large sub-grain is shown in Fig. 4f.

In the aged conditions, the recrystallized and recovered microstructure remains essentially the same as in the solution-treated condition.

3.4. Effect of the extrusion temperature and ratio on the as-extruded, solution-treated and aged mechanical properties

Fig. 5 shows the variation of the UTS (MPa), 0.2%PS (MPa), %EL and the short-rod fracture toughness, K_{IcSR} (MPa m$^{1/2}$) with the extrusion temperature in the AE, ST, UA, PA and OA conditions. In the AE condition, both the UTS and the 0.2%PS decreased as the extrusion temperature increased. The %EL increased marginally and K_{IcSR} was almost unaffected by the increase in the extrusion temperature. These results are consistent with the microstructural observations made earlier. It was observed that the degree of recrystallization in the interior of the material increased with the increase in the extrusion temperature and that this leads to decreased strength and increased ductility. In addition, the sub-grain size also increased with the increase in the extrusion temperature. This will also result in a lower strength at the higher extrusion temperatures. The recrystallized grains at the surface of the extrudate were also observed to be larger at the higher extrusion temperatures, which has the effect of further decreasing the strength of the extruded rod with the increase in temperature. However, the microstructure at the surface of the extrudate will have no influence on the mechanical properties of the tensile specimens tested in the present investigation as the surface of the extrudate was machined off in the process of the making of these specimens.

With solution treatment, the UTS and the 0.2%PS decreased, the %EL increased and the K_{IcSR} remained almost the same. This can also be explained on the basis of further recrystallization taking place with the solution treatment, as was observed in Fig. 3. However, the changes in the mechanical properties associated with the solution treatment in the case of the 525 °C extrudates were much less than those extruded either at 350 °C or at 400 °C. The reason for this is that the 525 °C extrusion temperature is already very close to the solution-treatment temperature, i.e. 530 °C, so that the extruded microstructure was relatively stable during the solution treatment. It also appears that when restoration occurs only dynamically during extrusion (i.e. at 525 °C), rather than partially dynamically and partially statically during extrusion at lower temperatures and solution treatment, the strength is increased, as the higher UTS value in the ST condition for the 525 °C extrudate would suggest. Thus, in the solution-treated condition, the extrusion temperature did not indicate a simple trend in its influence on the mechanical properties.

In the aged conditions, the strength is considerably increased and the %EL decreased due to the precipitation of the δ′-precipitates. K_{IcSR} drops sharply from the UA to the PA and OA conditions due to the growth of the δ′-precipitates, the equilibrium precipitates and the precipitate-free zones. However, the variation in the mechanical properties with the extrusion temperature remains roughly the same as in the ST condition.

The effect of the extrusion ratio on the mechanical properties in the AE, ST, UA, PA and OA conditions is presented in Fig. 6 for extrusion at 400 °C with a 3 mm s^{-1} ram speed. In all the conditions, the strength properties were higher, the %EL slightly lower and the K_{IcSR} almost the same for the lower extrusion ratio, 20:1. Again, this trend can be explained on the basis of the lower recrystallization observed at the lower extrusion ratio in Fig. 2.

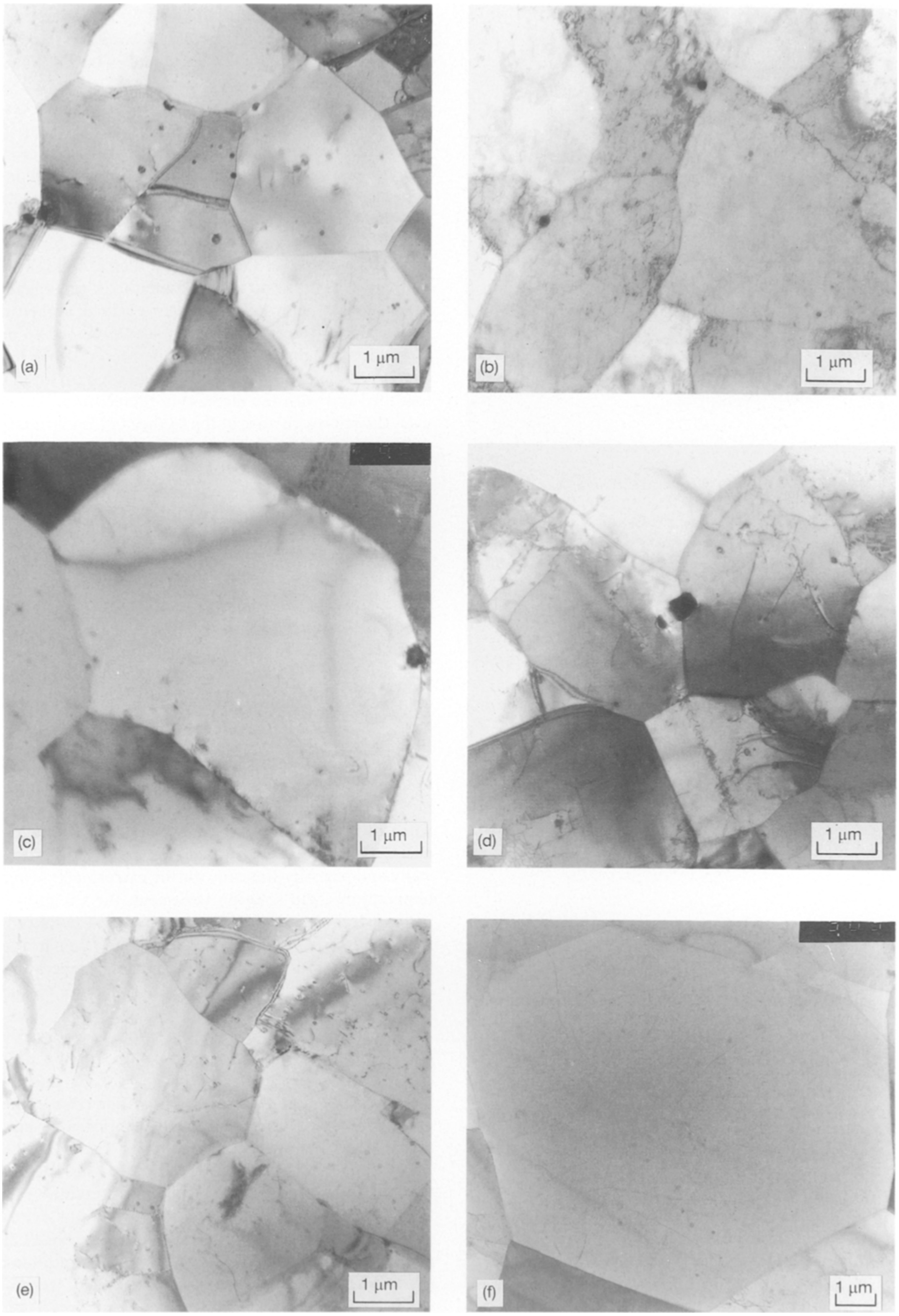

Figure 4 TEM showing the sub-grain sizes in the alloy: (a) extruded at 350 °C, at a 30 : 1 ratio, in the as-extruded condition, (b) extruded at 400 °C, at a 30 : 1 ratio, in the as-extruded condition, (c) extruded at 525 °C, at a 30 : 1 ratio, in the as-extruded condition, (d) extruded at 400 °C, at a 20 : 1 ratio, in the as-extruded condition, (e) extruded at 400 °C, at a 30 : 1 ratio, in the solution-treated condition, and (f) the same as (e) showing a large sub-grain.

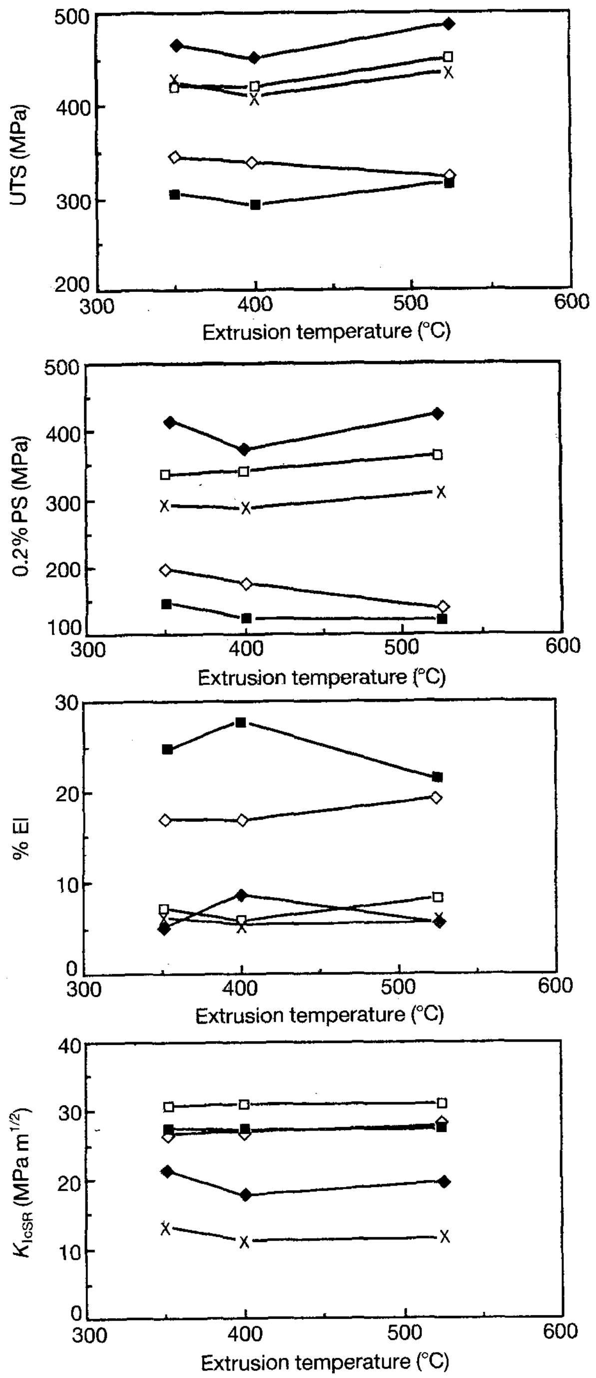

Figure 5 Variation of the mechanical properties with the extrusion temperature: (–◇–) AE, (–■–) ST, (–◻–) UA, (–◆–) PA, and (–×–) OA.

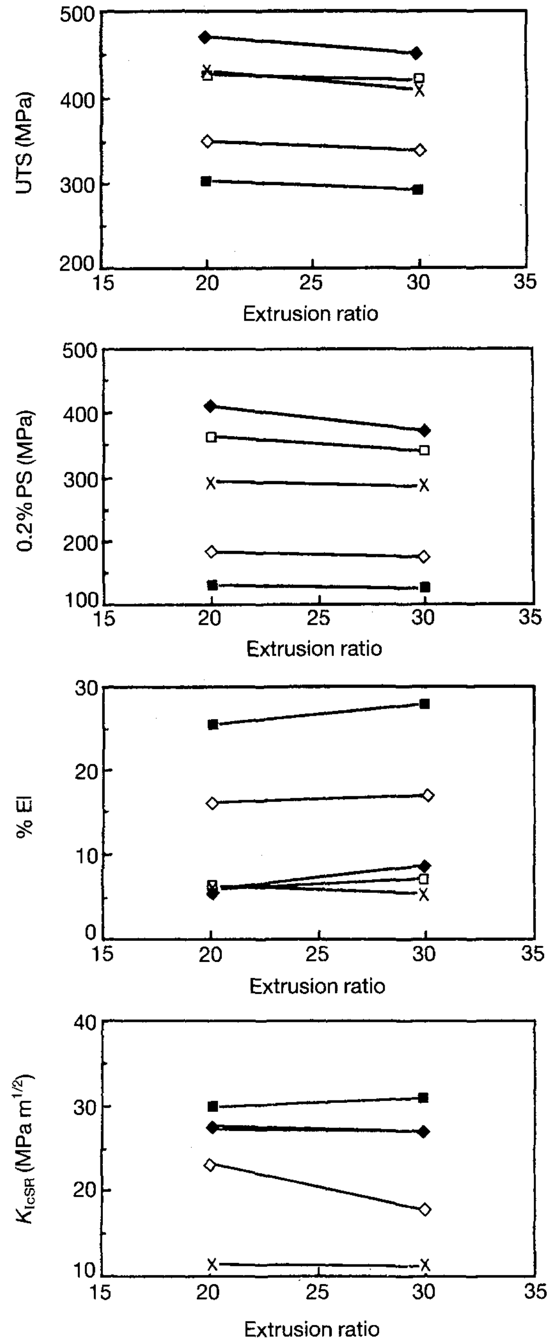

Figure 6 Variation of the mechanical properties with the extrusion ratio: (–◇–) AE, (–■–) ST, (–◻–) UA, (–◆–) PA, and (–×–) OA.

4. Conclusion

The surface of the extrudate is more recrystallized than the interior due to the higher strain, higher temperature and a loss of Li at the surface. All of these factors promote recrystallization.

An increase in extrusion temperature and ratio increases the degree of recrystallization in the extrudate. An increase in the extrusion temperature also increases the sub-grain size, whereas the extrusion ratio does not affect the sub-grain size. With the solution treatment, the growth of the already existing recrystallized grains and the nucleation of some new recrystallized grains takes place, whereas the sub-grain size of the recovered regions remains essentially the same as with the solution treatment.

The strength of the extrudates decreases and the ductility increases in the as-extruded condition with increases in the extrusion temperature and the extrusion ratio. The solution treatment decreases the strength and increases the ductility. The fracture toughness remains almost unaffected either by variation of the extrusion temperature and ratio or with solution treatment. All these trends can be explained on the basis of the degree of recrystallization observed in the interior of the extrudates: the higher the extrusion temperature, the higher are the extrusion ratio and the solution treatment, producing more recrystallization, and thus reducing the strength and increasing the ductility of the extrudates. The decrease in the strength with increases in the extrusion temperature is also caused by an increase in the sub-grain size with increases in the extrusion temperature. However, in the solution-treated condition, although the effect of the extrusion ratio on the mechanical properties is found to be the same as in the as-extruded condition, the trend with the extrusion temperature changes, and the extrusion temperature does not exhibit a simple trend in its influence on the mechanical properties. The reason for this change in the trend is not very clear. In the aged conditions, the recovered and recrystallized microstructures, as well as the effect of the extrusion temperature and ratio on the mechanical properties, remains roughly the same as in the solution-treated condition.

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