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Fabrication, properties and structure of a high temperature light alloy composite

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Particulate alumina reinforced Al–4Cr–1 Fe alloys were fabricated from rapidly solidified aluminium alloy powder and commercially purchased alumina powder by traditional powder metallurgical techniques involving powder mixing and cold compaction followed by hot extrusion. The tensile tests at ambient temperature indicated a considerable improvement in the mechanical strength at the expense of ductility and modulus. Poor values of modulus were explained by the presence of porosity in the composites. The high temperature mechanical properties of the matrix, tested at 350 °C after prolonged exposure to the test temperature under static air conditions, were intrinsically poor. Additions of the filler material, alumina particles, up to a weight fraction of 15% did not improve the high temperature performance of the matrix substantially. Possible causes for this are discussed and alternatives proposed.

1. Introduction

During the last decade efforts have been directed to develop an aluminium alloy as a possible replacement for titanium on specific strength basis for lightweight applications in the working temperature range between 250 and 350 °C. Two alloy systems based on Al–Fe and Al–Cr have been identified to fulfil the requirements. Aided by recent advances in rapid solidification technology, the production of concentrated aluminium alloys bearing transition metal elements with a fine microstructure have become a possibility; these are otherwise unobtainable by conventional technology. The parent materials, especially the atomized powders, comprise a spectrum of microstructures ranging from segregation free, otherwise known as partitionless morphology or microcellular morphologies in the very fine powders, to segregated cellular and/or dendritic microstructure in coarser powders.

There are still certain problems associated with these alloy systems, preventing their possible industrial applications. Especially at temperatures greater than 250 °C the tensile strength falls rapidly, attributed to the structural coarsening of densely populated dispersoids. The inherent characteristics of the Al–Fe and Al–Cr alloy systems as well their fabrication properties, (i.e. alloys based on Al–Fe system are inherently harder and difficult to process, whereas alloys based on Al–Cr are easier to fabricate and are comparatively softer), stimulated the development of certain hybrid compositions based on the Al–Cr–Fe ternary system, which were expected to obtain the best properties of both systems. Hence, an Al–4Cr–1Fe alloy has been primarily developed whose microstructure and properties at ambient temperature have been reported [1]. The mechanical properties of this material at high temperature has been found to be inferior to the parent binary systems. In order to improve the modulus and the high temperature performance of the Al–4Cr–1Fe alloy, various weight fractions of inert crystalline Al_2O_3 particles were mechanically incorporated at the expense of density. In this work, the extrusion characteristics and the properties at room and elevated temperatures of the composite alloy are presented and discussed.

2. Experimental procedure

The basic alloy, supplied by M/S Metalloys Ltd UK, was in the form of inert gas atomized powder with a median particle size of 35 μm and the particle size spectrum ranging from 8 to 110 μm. The morphology of the powder was predominantly spherical. The reinforcement powder, supplied by Buehlers UK, was the polishing grade 5 μm size Al_2O_3 powder with predominantly acicular morphology.

The as-atomized powder was divided into two batches. The first half of the powder was split into 600 g batches, which were subsequently cold compacted in a double action press at a pressure of 500 MPa and hot extruded in a 5 MN vertical extrusion press. The extrusions were carried out with a flat faced 20:1 die at various temperatures ranging from 400 to 550 °C and the extrudates were press quenched. The other half of the metal powder was divided into three batches, each of which was separately mixed with various weight fractions (5, 10, and 15 wt %) of Al_2O_3, to yield finally batches of 600 g. Prior to mixing the Al_2O_3 powder was dried at 500 °C for 24 h. The mixing was carried out in a proprietary mixer for 40 min. The mixed powders were subsequently cold compacted and hot extruded at 500 °C. The composite billets were coated with colloidal graphite prior to extrusion to avoid surface cracking.

The assessment of the microstructure was carried out by optical and transmission electron microscopy. Samples for TEM were sectioned in the transverse plane to the extrusion direction and prepared by a conventional electropolishing technique. Static mechanical properties were measured on roundbar tensile samples, Hounsfield No. 13, which were machined parallel to the longitudinal direction. The tests were carried out both at ambient temperature and at 350 °C at a strain rate of $5 \times 10^{-4}\,\mathrm{sec}^{-1}$. The specimens tested at 350 °C were preheated at this temperature for 100 h.

3. Results and discussion

3.1. Extrusion characteristics

Hot extrusion is the principal consolidation stage in the present series of experiments. The extrusion behaviour of the matrix alloy has been reported in a previous publication [1]. In the present series of experiments all billets were preheated in the induction heater for a short time possible to avoid any precipitation and coarsening reactions. The billet containing 15 wt % of alumina could not, however, be heated in the induction heater and hence an air circulation furnace was used. This difficulty of preheating in the induction furnace could be attributed to the low green density of the compact and increased resistivity presented by the Al_2O_3.

The breakthrough pressure as a function of the reinforcement content is shown in Fig. 1a. The peak pressure for extrusion increases non-linearly from 670 to 940 MPa with increase in the weight fraction of alumina from 0 to 15%. A similar trend was observed in the case of the steady state pressure, though the peak pressure increment (ΔP), defined as the difference between the peak and the steady state pressures, initially decreases by 40 N mm^{-2} from 0 to 5% reinforcement of alumina content but increases beyond this, see Fig. 1b. The increase in the breakthrough pressure with increasing alumina weight fraction can be explained on the basis that the presence of coarse second-phase particles in a soft matrix increases the flow stress of the matrix and consequently the pressure, however, the increased peak pressure requirements to extrude the unreinforced billet compared with unreinforced billets, maybe attributed to a frictional effect, because the unreinforced billets were extruded unlubricated.

3.2. Microstructural characteristics of the extrudates

Microstructural analysis of the extruded matrix material as a function of the extrusion parameters has already been reported [1]. The distribution of the alumina particles in the extrudates of 5, 10 and 15% Al_2O_3 content are illustrated in Figs 2a to b, c to d, and e to f, for the longitudinal and transverse directions, respectively. It is evident that, even though the distribution of the alumina particles appears homogeneous in the transverse direction especially with decreasing weight fraction of alumina, in the longitudinal direction the alumina particles are aligned along the extrusion direction forming semi-continuous stringers, thus the microstructure is composed of elongated bands of macroscopically filler free zones separated by alumina particle stringers. These zones between stringers are obviously the original individual metal powder particles welded and extended in the extrusion direction. Another characteristic of the composites containing a higher fraction of alumina is the presence of porosity, the presence of pores appears as dark spots in the micrographs, i.e. see arrowed example in Fig. 2f. The formation of the porosity can easily be explained by analysing the distribution of the reinforcing particles in the cold compact prior to extrusion. One factor affecting the porosity is the ratio of the average metal powder particle size to the alumina powder particle size. When this ratio is high every alloy powder particle will be uniformly coated with fine alumina particles, termed powder aggregates, during mixing. Hence, as illustrated in the schematic diagram in Fig. 3, during the extrusion process and within the aggregates the metal-to-metal welding is restricted in regions in contact, because firstly the presence of the alumina particle prevents metal–metal contact and secondly the material shears along preferential planes. This results in unfilled volume. The effect becomes more predominant with increasing the weight fraction of the alumina. Consequently, microstructures with pores result in poor interparticle welding and hence poor mechanical properties.

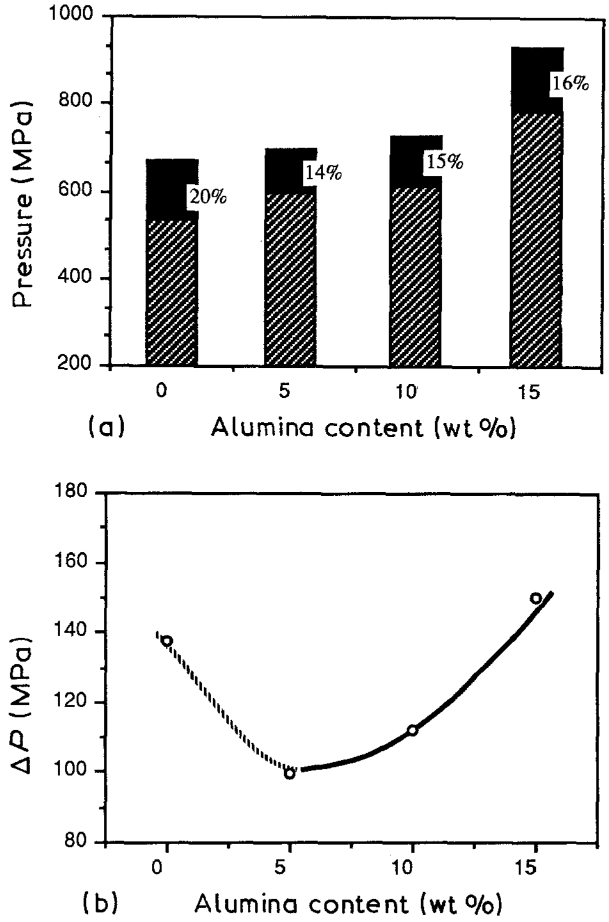

Figure 1 Affect of the reinforcement content on (a) the maximum (■) and steady state (▨) extrusion pressure, and (b) the peak increment.

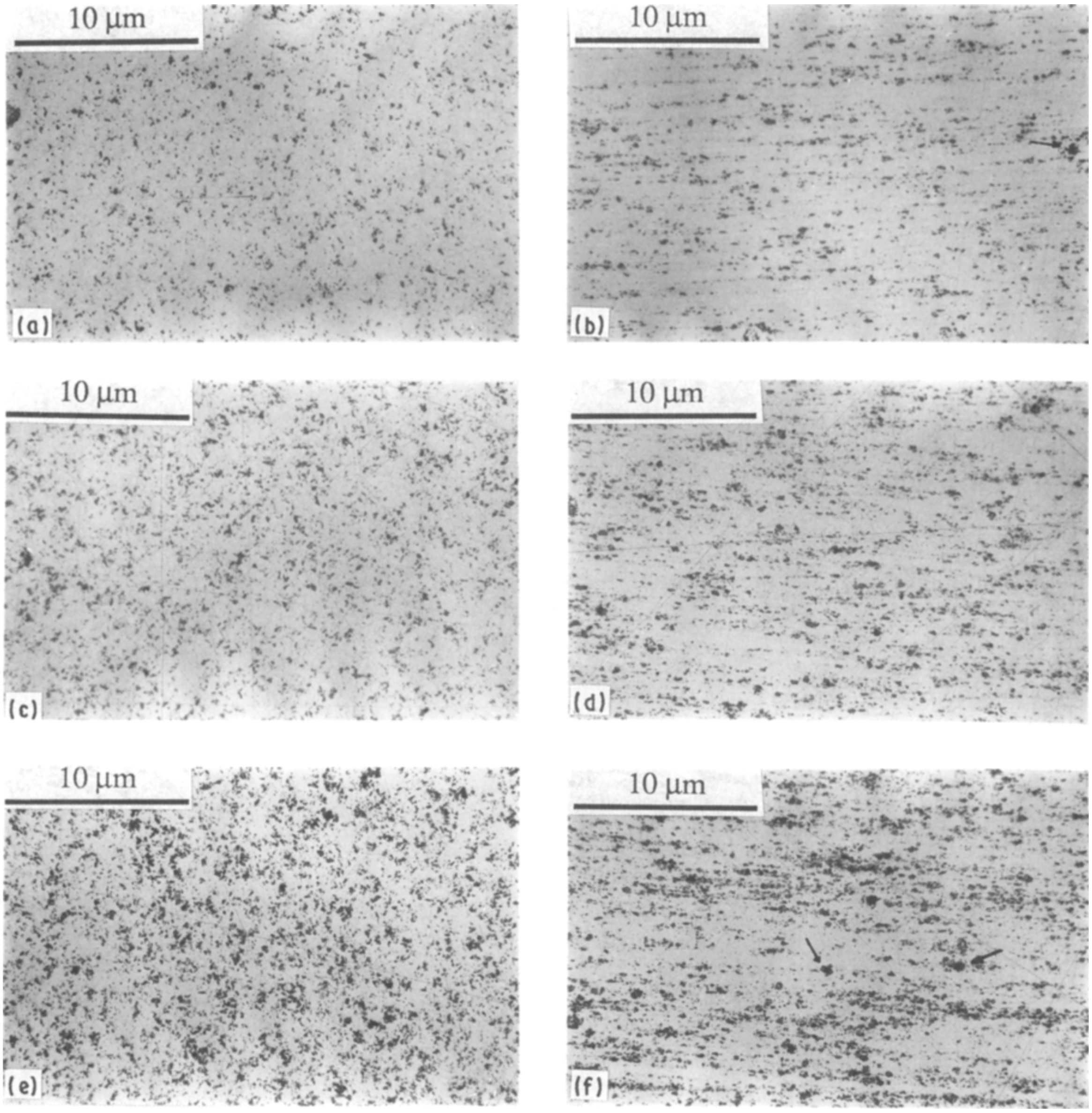

Figure 2 Optical microstructures along the longitudinal and transverse direction of the (a), (b) 5 wt %, (c), (d) 10 wt % and (e), (f) 15 wt % Al_2O_3 composite alloys, respectively.

The microstructural heterogeneity attributed to the porosity is supplemented by the microstructural heterogeneity of the matrix material. The matrix microstructure of the as-extruded material contains predominantly subgrains modified by the presence of the matrix intermetallic particles–dispersoids. The deformation mechanism is controlled by the rate of dynamic recovery, dependent upon the interaction between the moving dislocation and the matrix dispersoids. The consequence of this interaction is that in some areas the substructure is coarser and in some other areas very fine, governed by the local distribution of the dispersoids, whose size and distribution depends upon the original metal powder particle size. Thus in general large powder particles contain a coarser distribution of dispersoids and hence coarser subgrains whereas the finer particles inherit a finer substructure. This inhomogeneity also contributes to the mechanical property modifications. The presence of the reinforcing alumina particle along the metal particle boundary does not modify the matrix microstructure drastically, see Fig. 4. It is also interesting to note that in this alloy the incidence of undeformed fine metal powder particles is minimal even at lower extrusion temperature in contrast to an *R/S* Al–Fe alloy [2], which could be attributed to the fact that Al–Cr alloys are softer than Al–Fe alloys.

3.3. Mechanical properties

The major mechanical properties of the as-extruded material have been assessed at ambient and high temperatures in terms of 0.2% proof stress, ultimate tensile stress, fracture strain and specific strength properties. The mechanical properties at ambient temperature are shown in Fig. 5a to c. The proof stress and the ultimate tensile stress increase considerably with increase in the weight fraction of alumina content, see Fig. 5a, but the increase in strength is accompanied by a drastic reduction in the tensile ductility. The reader should note that the weight fraction of the alumina content does not include the submicrometre

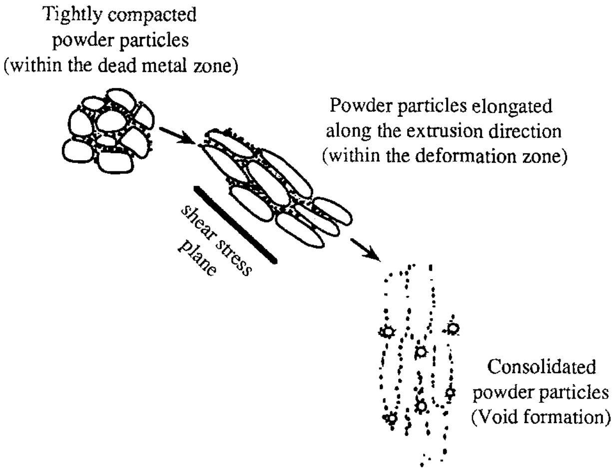

Figure 3 Schematic representation of pore formation during extrusion.

aluminium oxide particles dispersed within the matrix, originating from the fragmentation of the oxide layer surrounding the powder particles, which was formed during atomization.

Two factors affecting the plastic flow and thus contributing to the strengthening of the basic aluminium matrix arise from the matrix dispersoids and the reinforcing alumina particles. The mechanism of strengthening in the case of the matrix dispersoids can be easily explained by the classical Orowan mechanism. The contribution of the alumina particles cannot, however, in general be described by the classical theories of dispersion hardening based on the Orowan model. The increase in the flow stress of the matrix in such case can be explained however on the basis of *geometrically necessary dislocations* to accommodate the strain difference between the matrix and the reinforcement, because when the powder aggregates deform the harder phase i.e. the alumina particles, deform firstly elastically. In order to describe the flow behaviour of the aggregate, the stress and the strain distribution in both phases must be known at a given total strain. Two simple models based on *equal-stress* and *equal-strain* distribution can give some ideas of the influence of the hard constituent on the flow stress of the matrix. The real partitioning of the stress will obviously lie in between the two models [3]. Based on this model the strength of the aggregate for a given percentage of the hard constituent increases to a maximum with increase in the size of the particles and then drops with further increase in the size. This is primarily because if the particles are below a certain critical size, the load transfer to the particles are not efficient and the particles do not deform sufficiently to contribute to the total deformation in presence of the matrix. For a given particle size an increase in the percentage of the harder constituent requires a greater dislocation density to accommodate the strain difference. The mean free path of the dislocations before meeting the obstacles also decreases because the stress fields associated with the particles overlap resulting in considerable resistance to dislocation motion. This results in the increase in the strength of the aggregate with increase in the particle content.

In the above discussion it is assumed that the aggregate contains no porosity. In a real situation the material contains a certain amount of porosity. This is clear from the density measurements as illustrated in Fig. 5b, and the micrographs in Fig. 3a to f. The results indicate that the porosity increases with increasing the weight fraction of alumina, see Fig. 5b. It is well established that the porosity reduces the tensile strength [4]. This is accomplished by effectively

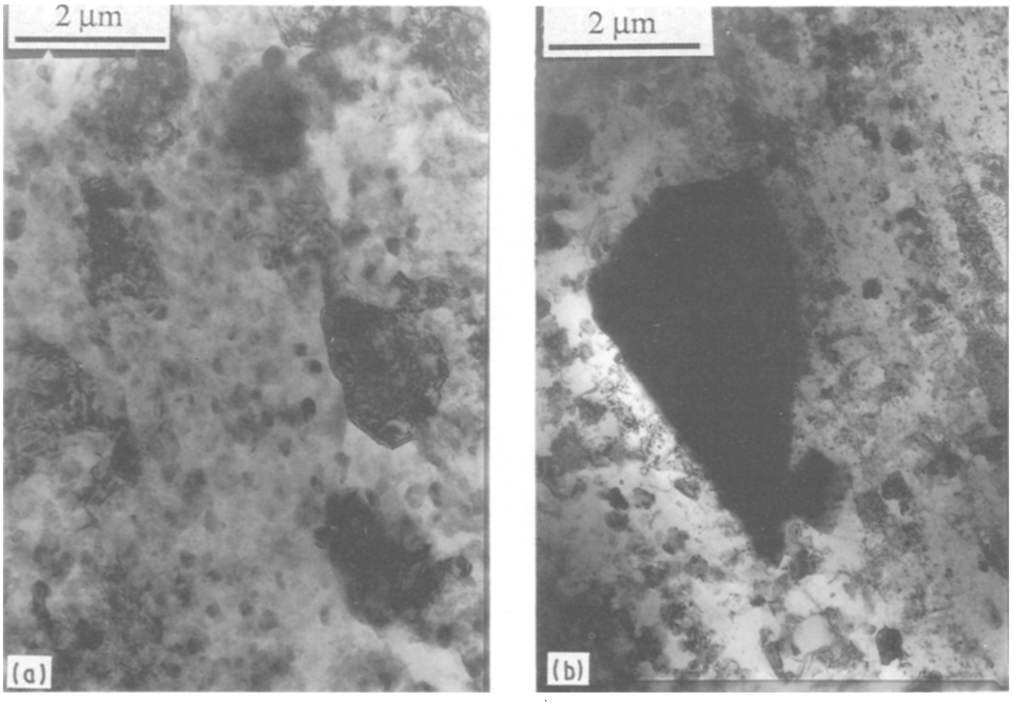

Figure 4 HVEM microstructures showing microstructural heterogeneity attributed to both the inherent as-atomized microstructure and the presence of the reinforced alumina particles: (a) coarse spherical chromium rich precipitates, and (b) fine band in contact with a relatively large alumina particle.

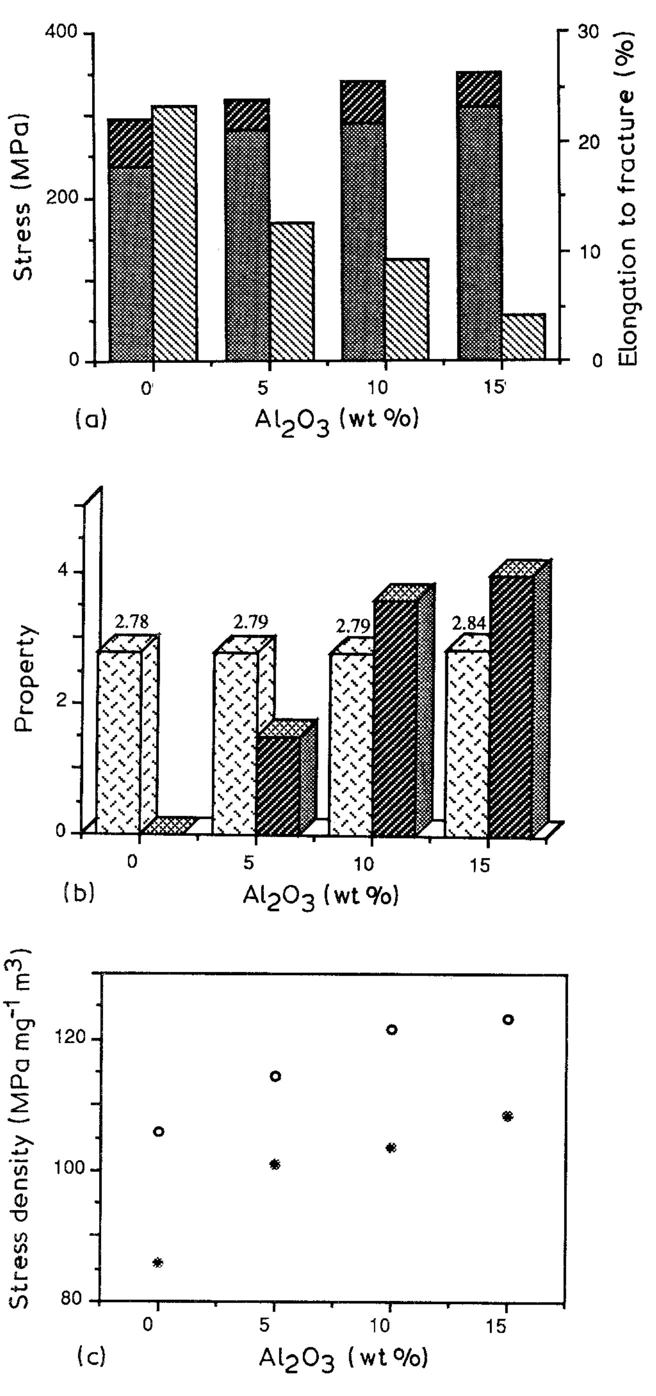

Figure 5 Variation of the mechanical properties as a function of the alumina content: (a) 0.2% proof stress, (▦), UTS (▨) and elongation (▧) to fracture, (b) density (▦) and porosity (▨) and (c) specific UTS (○) and specific proof stress (●).

reducing the local load-bearing capacity of the material due to the stress concentration near the pores, and also by reducing the load bearing cross-section area, thus the strength levels obtained in the present case, although increasing with the reinforcement content, are lower than the theoretically expected mean strength values.

In addition, it is implicitly assumed that the stress transfer from the matrix to the reinforcement occurs efficiently and completely. This requires that the interface is strong and does not delaminate due to poor bonding along the interface. In a real situation the interface shear strength is not very high and can result in slipping and delamination along the interface. This is clearly illustrated in Fig. 6. The fractographs, at higher magnification, show that fracture occurs by void nucleation and growth around the matrix particles, while the reinforcing particles along the boundary delaminate. The final fracture occurs by the link up of the voids leading to a dimpled fracture surface. It is evident that the fracture surface contains two sets of dimples. The first set of dimples, which are small and more spherical, is associated with the matrix particles. The second set is related to the reinforcing alumina particles and is coarse as marked (*) in Fig. 6b. The marked area in Fig. 6b, is very smooth and is an indication of delamination along the interface due to low interface shear strength. With increase in the percentage of reinforcement the incidence of delamination occurs more frequently. This also facilitates secondary cracking along the direction of extrusion and along the powder particle boundaries. In some rare cases, where the alumina particle size is large, the particle itself fractures before the interface delaminates. This is shown schematically in Fig. 7. This indicates that coarser reinforcing particles undergo considerable deformation before the aggregate fractures. A typical example is shown in Fig. 6e. The particle at the centre of the field, which is approximately 10 μm, has fractured into three parts and the fracture surface of the debris show some shear marking.

Table I shows that the tensile modulus increases initially with increase in the reinforcement content up to 5% and then drops systematically with further increase in the alumina content. This correlates well with the increase in the porosity in the microstructure with increase in the percentage of alumina. Treating the porosity as due to isolated closed pores as a first approximation as inclusions with zero tensile modulus, it becomes clear that the modulus of the aggregate containing porosity will be lower than the aggregates containing no porosity, thus in the present case the increase in the modulus of composites due to the presence of hard particles is offset by the porosity by an amount proportional only to the percentage of the porosity in the microstructure.

The influence of tensile test temperature on the mechanical properties is shown in Fig. 8a and b for both the matrix and the composite materials respectively. The mechanical properties are far lower than the expected values in the matrix alone. There is a slight increase, however, with a decrease in the extrusion temperature. There is also a reduction in the ductility values with a decrease in the extrusion temperature. The poor strength of Al–Cr alloys becomes obvious comparing the propensity for microstructural coarsening with other alloys, such as Al–Zr, Al–Fe,

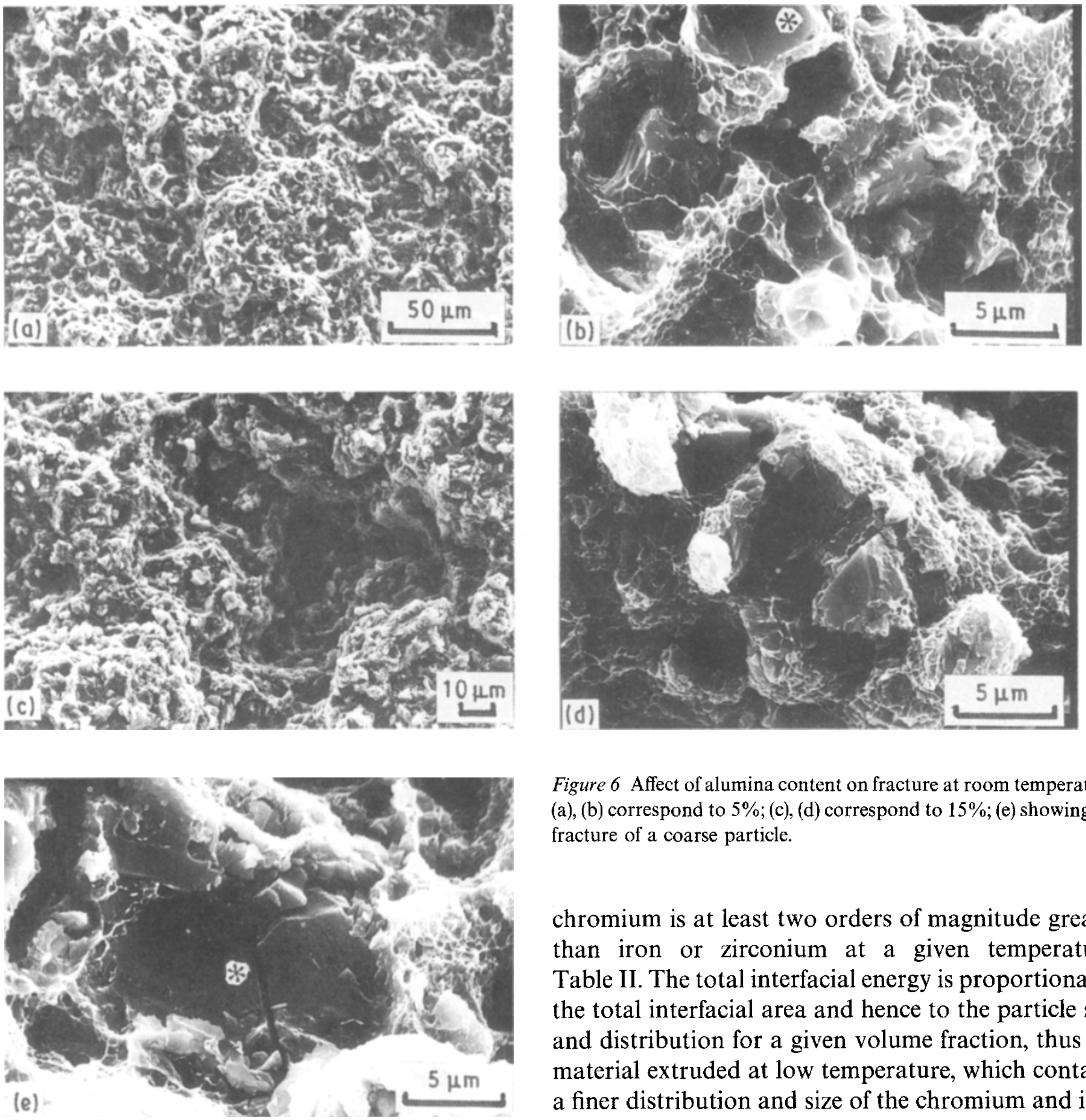

Figure 6 Affect of alumina content on fracture at room temperature. (a), (b) correspond to 5%; (c), (d) correspond to 15%; (e) showing the fracture of a coarse particle.

and Al–Ti, hence it is necessary to understand the factors contributing to the coarsening. The primary driving force for coarsening is the reduction in the interfacial free energy, thus the coarsening rate is governed by the total interfacial energy, the temperature, and the diffusivity of the chemical species constituting the second phase particles. The diffusivity of chromium is at least two orders of magnitude greater than iron or zirconium at a given temperature, Table II. The total interfacial energy is proportional to the total interfacial area and hence to the particle size and distribution for a given volume fraction, thus the material extruded at low temperature, which contains a finer distribution and size of the chromium and iron bearing dispersoids, would be expected to have higher coarsening rate than the material extruded at high temperature. In spite of this, the material extruded at low temperature contains at the end of heat treatment finer distribution of the particles compared to the materials extruded at high temperature. This accounts for the marginal increase in the strength of the materials extruded at low temperature after heat treatment.

TABLE I Effect of the alumina content on the modulus of the composite alloy

%wt Al_2O_3	0	5	10	15
Modulus (GPa)	81.75	92.52	88.15	86.88

Also shown in the Fig. 8b are the affect of test temperature on the strength of the composites containing different volume fractions of alumina. It can be seen that the addition of alumina does not improve the high temperature strength of the matrix substantially. This is in contrast to the behaviour of SiC reinforced composites [2]. This can be explained on the basis that at high temperature the dynamic recovery in the

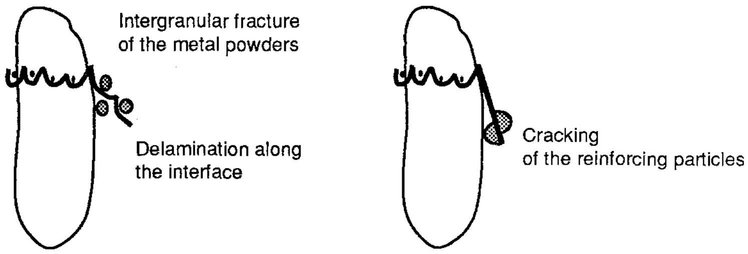

Figure 7 Schematic representation of the fracture mechanism.

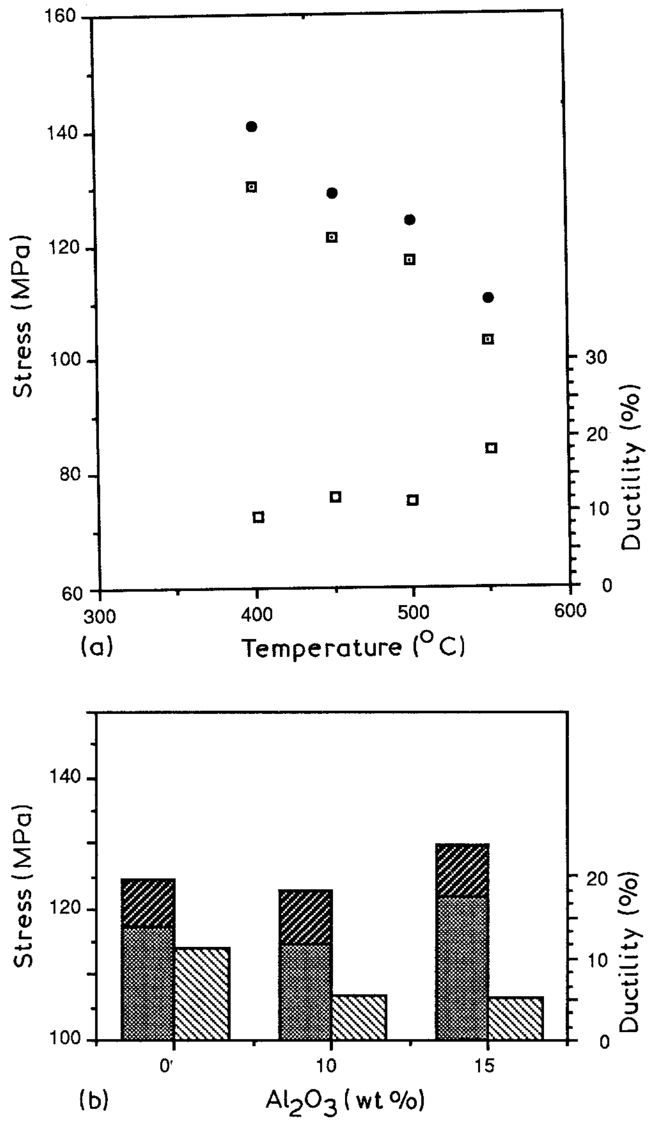

Figure 8 Affect of the tensile test temperature (a) and alumina content (b) on the mechanical properties of the non-reinforced Al–4Cr–1Fe alloy and 550 °C extruded composite material, respectively. (⊡ ▩ 0.2% proof stress, ● ▨ UTS, □ ▧ ductility.)

matrix offsets the strengthening effect due to the geometrically necessary dislocations to accommodate the strain difference between the matrix and the reinforcement. This effect combined with the influence of porosity on the mechanical properties results in poor strength of the composites. It is possible to improve the strength however by increasing the weight fraction of the reinforcement. The fracture surface of the high temperature tensile tested samples are shown in Fig. 9. The fracture surfaces are quite different when compared to the room temperature fracture. The fracture surface still contains dimples, but with rounded edges typical of a high temperature fracture. The fracture surface of the composites show that delamination along the interface between the alumina particle and the matrix is still an integral feature of the fracture at high temperature.

TABLE II Diffusivity of some transition elements in solid aluminium

Element	D (cm^2 sec^{-1})
Fe	5.4×10^{-14}
Zr	3.4×10^{-18}
Cr	1.1×10^{-12}
Ce	8.4×10^{-16}
Ti	3.0×10^{-16}

4. Conclusions

Extrusion processing of alumina particulate reinforced Al–4Cr–1Fe has been carried out and the pressure required to extrude the aggregate increases with increase in the alumina content leading to a restriction in the lower limit of the temperature of extrusion. Consolidation takes place during the hot extrusion stage yielding a microstructure consisting of elongated metal powder particles in the extrusion direction bounded by the stringers of the alumina particles along the powder particle boundary. At higher percentage of the reinforcement the microstructure also contains a certain amount of porosity related to the agglomeration of the alumina particles.

There are considerable improvements in the ambient temperature strength with increase in the amount of alumina content. This is accompanied by a reduction in the ductility of the matrix and the tensile modulus. The reason is the porosity of the structure, it should be possible to improve the properties by subjecting the material to a secondary metal forming operation in which the force is applied to in the direction transverse to the extrusion direction. This enables the matrix to flow through the porosity and fill the pore volume.

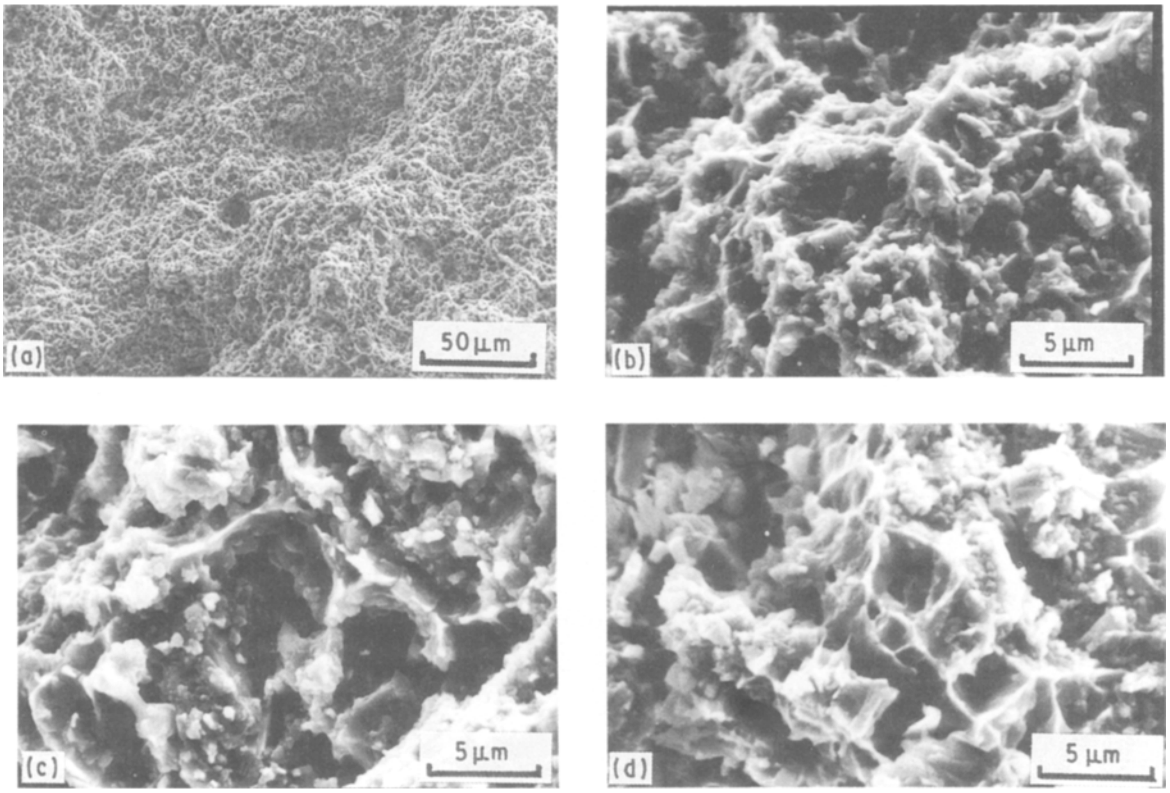

Figure 9 Affect of tensile test temperature on the fracture path of 550 °C extrudes. (a), (b) matrix with no reinforcement; (c), (d) composite with 15% alumina.

The matrix has intrinsically poor high temperature strength. This is inevitably due to the coarse size and distribution of the dispersoids in the matrix. The mechanical properties of the composites at high temperatures are also disappointingly low. Analysis of the contribution to the strength from the reinforcements indicate that an increase in the volume fraction combined with a reduction in the structural porosity by changing the processing conditions, can improve the properties considerably. Also changing the filler material from alumina to materials which are intrinsically harder, such as Tic or SiC, should benefit the matrix strengthening.

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