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Kinetics of Carbonisation, Microstructure and Properties of Al-Al_4C_3 System Prepared by Mechanical Alloying

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Abstract

The method of mechanical alloying process is described. Carbon transformation to carbide Al_4C_3 is characterised within different heat treatment schedules and nine commercial carbon powders are tested. The micromechanism of carbon incorporation into the metallic powder, and the compacting of it are described. The influence of dispersed carbides on mechanical properties is evaluated together with the influence of deformation on microstructure and properties.

Keywords: aluminium-graphite powder system, mechanical alloying, carbon to Al_4C_3 transformation, microstructure, mechanical properties

Introduction

Mechanical alloying was first developed and used to prepare superalloys with a nickel matrix, and the method spread to other alloys later. Dispersoids can be formed in a solid state reaction by introducing materials that react with the matrix in the time following heat treatment.[1-2] A mode of mechanical alloying is reaction milling, developed for dispersion strengthened aluminium produc - tion. [3-4]

The aim of this paper is to study the influence of the various graphite types when mixed with Al powder, and heat treatment procedure on the microstructure and properties of dispersion strengthened aluminium type Al-Al_4C_3.

Experimental Material and Methods

The experimental material - dispersion strengthened aluminium with Al_4C_3 particles, was prepared by intense milling of aluminium powder with different types of carbon, as shown in Table1. The prime aluminium powder grain size was 100 μm with the carbon content of 0.6 - 3 wt. %.

The aluminium composite dispersion strengthened by Al_4C_3 particles has been prepared by the method of mechanical alloying. The milling time used in all tested specimens was 90 min. The final carbide content was in the range of 2.5 - 12 vol.%. The obtained mixture was compacted at 600 MPa and heat treated at 450, 500, 550, and 600°C whereas treatment times of 1, 3, 10, and 30 hours were employed. The final compacting by hot extrusion at a temperature of 550°C and a reduction of 94% in the cross circular section of extruded bars was applied[5]. The experimental material-has been both prepared, and tested by gas chromatography for carbides Al_4C_3 content, at the Institute for Chemical Technology of Inorganic Materials, TU Vienna.

Table 1. Types of different carbon types used

Notation	Type	Commercial Carbon	Notation	Type	Commercial Carbon
A	a_1	LTD	F	a_2	Farbruss FW 2
B	a_1	Spezialschwarz 5	G	a_2	Flammruss 101
C	a_1	Spezialschwarz 500	H	c	Thermax
D	a_1	Printex 30	I	b	Grafit KS 2.5
E	a_2	Printex 400			

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Milling and Carbonisation Kinetics

Figure 1. shows the milling kinetics, the effect of milling time on the dislocation density and subgrain size of the matrix. After milling 120 min these parameters are constant.[(6)] From the results, the homogeneity of carbide distribution and contact surface area influence the Al+C transformation kinetics to Al_4C_3. The dependence of the transformation rate on temperature and holding time for the 4 carbon types is shown in Figure 2. The good susceptibility to transformation for porous furnace black (a_1 and a_2) and that of electrographite (b) is evident. The porous carbon types are incorporated into the matrix by friction during milling, the distribution is even, and clustering is small. On the other side, hard graphite (c), resists disintegration and the granules are large.

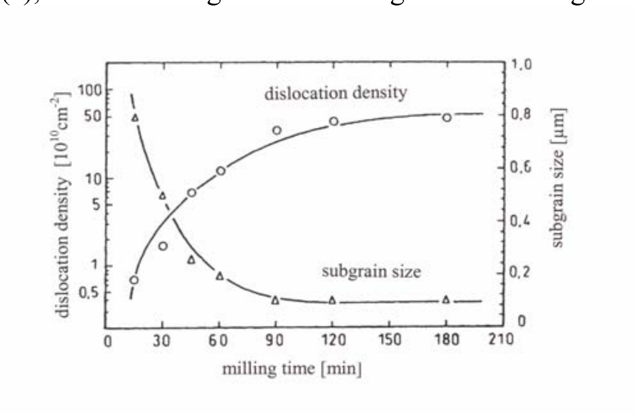

Figure 1. Dislocation density and subgrain size in Al + mass-% C milled granulate, as a function of the milling time

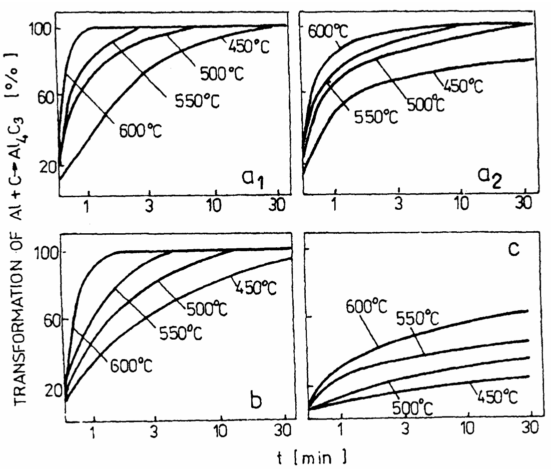

Figure 2. Dependence of carbon to the carbide transformation rate on heat treatment temperature and hold time for four carbon types

Microstructure and Mechanical Properties

Light microscopy microstructure analysis of the produced compacts proved a high homogeneity of dispersed particle distribution in the direction perpendicular to the direction of hot extrusion. In the longitudinal direction of the bar as a result of hot extrusion the Al_4C_3 carbide particles were arranged into bands.

The milling mechanism the of Al – 1C system from the time point of view is shown in Figure 3 – 6. Microstructure changes occurred in the fracture processes and welding of the particles with incorporation of C phase and forming of final granules.

Figure 3. State of granules after 10 min milling of Al + 1C material

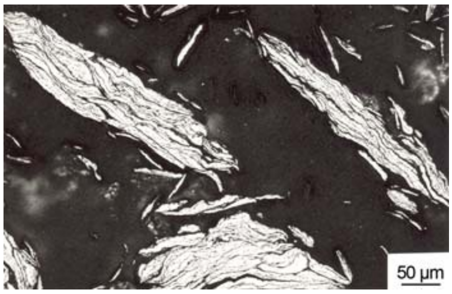

Figure 4. Granules after 30 min milling – welding process, Al+1C material

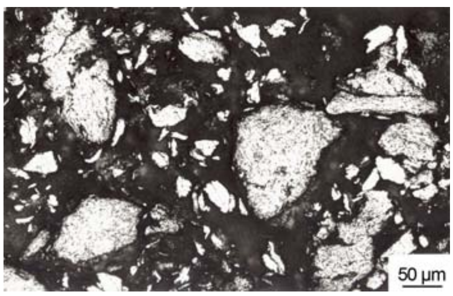

Figure 5. State of granules after 60 min milling, Al + 1C material

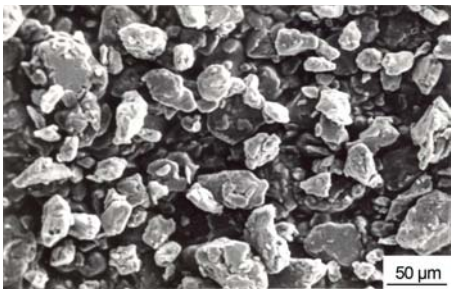

Figure 6. Shape of granules after 240 min milling of Al + 1C material

Electron microscopy analysis was conducted using carbon replicas and thin foils. TEM of thin foils offered better results. For all the tested carbon combinations from the A to I labels, thin foils were produced for the heat treatment 450°C/30 h. The Al_4C_3 particle size and the subgrain size were measured using the thin foils. The dispersed phase Al_4C_3 particle size was measured on 200 to 300 thin foil structures, and it was constant and as small as 30 nm. The particle size was influenced neither by the carbon type nor by the heat treatment technology applied. Subgrain size measured in the range of 100 grains in thin foils depended on the carbon type, as well. It ranged from 0.3 to 0.7 μm. The stability of properties, resulting from graphite type I (KS 2.5), led to the highest production and utilization of this type of dispersion strengthening.[7]

In our previous works.[8-10] we have evaluated the distance between the particles by point object simulation methods. This includes the mean interparticle distance λ_μ, the mean minimum distance λ_ρ, the mean visibility λ_v, and the mean free spherical contact distance λ_9. The characteristics and properties of these parameters have been analyzed in.[9]

During the last years, a new approach to the description of point systems has been developed intensively, which is referred to as polygonal methods.[10] The dual representation formed in the above way describes completely the given point system. Properties of Voronoi tessellation and their various generalizations are being very intensively studied now the state of this study is given in the monograph.[11] Intermediate stages of evaluation for thin foil (a), outlines of particles (b), and of reference points (c) are documented in Figure 7.

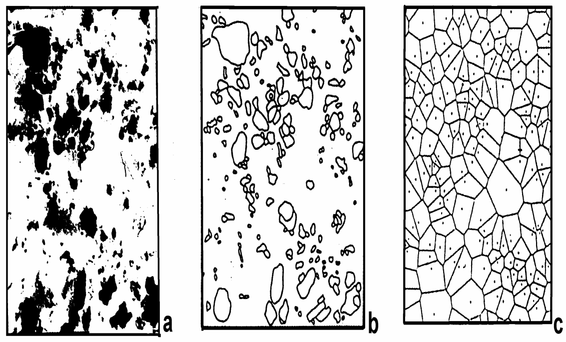

Figure 7. Intermediate stages of evaluation for this foil (a), outlines of particles (b) and reference points (c)

The Al-Al_4C_3 system with 4 vol. % of Al_4C_3 was tested under different tensile conditions, where three different strain rates and different testing temperatures up to 450ºC were used.[12-13] The results are shown in Figure 8. The deformation mechanism and fracture mechanism were analysed corresponding to different testing conditions. For the higher strain rates of $10^{-1}s^{-1}$ at 450°C, a significant growth of plastic properties was observed. The high uniform elongation A_5 of the specimen gauge length, and corresponding reduction values of the reduction in area Z were manifested in Figure 9. The ductility anomalies are showing an onset of a type of superplasticity. According to.[14-16] it was proved that for materials Al - 12Al_4C_3, the main mechanism responsible for superplastic behaviour is the grains rotation process and not sliding.

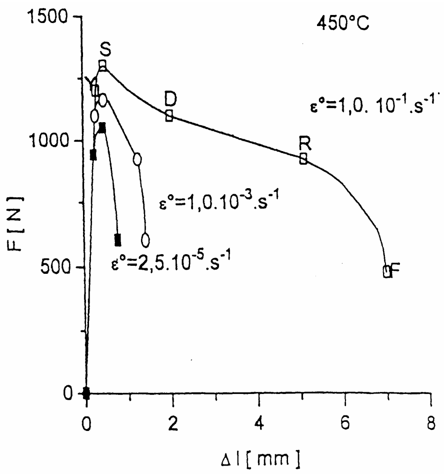

Figure 8. Stress-strain dependences at 450°C

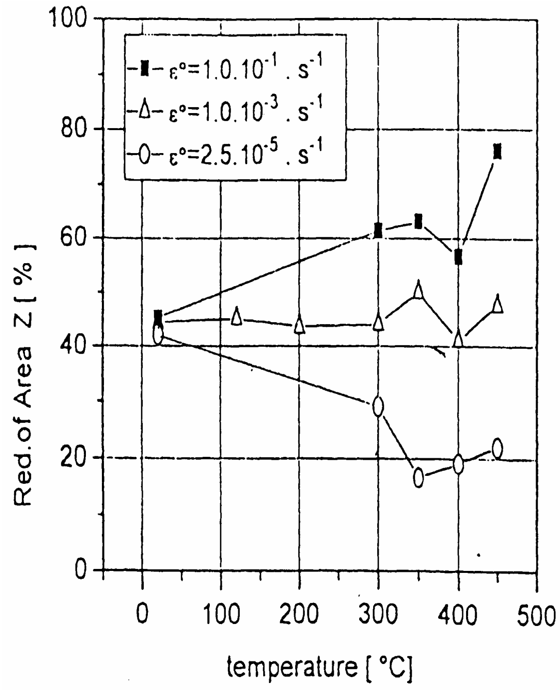

Figure 9. Reduction of area Z as a function of strain rate and temperature

Conclusion

Results can be summarized as follows:

1. It was shown that the transformation efficiency of carbon to Al_4C_3 by heat treatment of aluminium with the porous furnace black a) and electrographite b) is higher, than that of the hard cracked graphite c).
2. Microstructure changes consisted in the fracture processes and welding of the particles with incorporation of C phase and forming of final granules.
3. The dispersed phase Al_4C_3 particle size was measured on 200 to 300 thin foil structures, and it was constant and as small as 30 nm. The particle size was influenced neither by the carbon type nor by the heat treatment technology applied. Subgrain size measured in the range of 100 grains in thin foils depended on the carbon type, as well. It ranged from 0.3 to 0.7 μm.
4. The temperature dependencies of ductility, and reduction of area in temperature range of 350–450°C and strain rate of 10^{-1} s^{-1}, indicated a considerable increase of these properties. In case of the volume fraction of Al_4C_3 changes from lower to higher, the grain rotation mechanism dominates instead of the grain boundary sliding.

Acknowlegements

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