Microstructure Analysis of Carbonization Kinetics of Al-C System

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ABSTRACT

Dispersion strengthened aluminium compacts have been prepared by powder metallurgy. Microstructure is based on the aluminium matrix strengthened with dispersed Al_4C_3 particles. The strengthening is direct by dislocation movement retardation, and indirect by deformation induced microstructure modification in the next technological steps. The method of mechanical alloying process is described. Carbon transformation to carbide Al_4C_3 can be characterised with different heat treatment schedules and nine different types of commercial carbon powders.

Keywords: Mechanical alloying, C to Al_4C_3 transformation, Al-Al_4C_3 dispersion strengthened system, microstructure

1. INTRODUCTION

The aim of this paper is to describe the influence of the various graphite types and heat treatment procedure on microstructure and properties of dispersion strengthened aluminium alloys of Al-Al_4C_3. The effect of carbide characteristics on mechanical properties is evaluated together with the influence of deformation mode on microstructure development and mechanical properties /1-3/.

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2. MATERIALS AND EXPERIMENTAL METHODS

The experimental material - dispersion strengthened aluminium with Al_4C_3 carbide particles was prepared by intense milling of aluminium powder with different types of carbon, as shown in Table 1. The prime aluminium powder grain size was 100 μm with the carbon content of 3 mass %.

Table 1

Types of different used carbon types

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	Graphite KS 2,5
E	a_2	Printex 400			

Aluminum alloy dispersion strengthened by Al_4C_3 particles has been prepared by the method of mechanical alloying. The final carbide content was around 12 vol.%. The obtained mixture was compacted at 600 MPa and heat treatment at 723, 773, 823, and 873 K whereas treatment times 1, 3, 10, and 30 hours were employed. Final compacting by the hot extrusion at temperature of 823 K and reduction rate of 94 % on the cross section was applied /4/. The experimental material has been both prepared, and tested by gas chromatography for carbides Al_4C_3 content in the Institut für Chemische Technologie Anorganischer Stoffe TU Wien.

3. RESULTS AND DISCUSSION

The correlations between physical and chemical properties and milling parameters, or carbide transformation rate, and properties of the produced compacts were examined for nine samples with different types of commercial carbon labelled by A to I.

The different carbon types showed different distributions of carbon in aluminum powder. Their susceptibility to milling was measured by the ability to prepare homogeneous distribution without being formed clusters. According to the obtained results the carbon formed was divided into four types:

a_1) porous types of furnace black, made by incomplete burning of carbohydrates at low temperatures, with very good properties. They are fine, with high contact surface, and easy destruction of clusters.

a_2) porous types of furnace black, made by incomplete burning of carbohydrates at higher temperatures. They are fine, but they form more stable clusters, resistant to desintegration.

b) electrographite, with layered structure, with good susceptibility to milling, though coarse grained and with smaller contact surface; comparable to furnace black (a_1, and a_2).

c) cracked carbon, forms strong clusters, and the carbon to carbide transformation rate is low.

The milling kinetics is described in more detail in reference /5/. The homogeneity of carbide distribution and contact surface area influences the Al+C transformation kinetics to Al_4C_3. The dependence of Al+C to Al_4C_3 transformation rate on temperature and hold time for four carbon types is given in Fig.1. Good susceptibility to transformation of porous furnace black (a_1 and a_2) and that of electrographite (b) is found.

The percentage of carbon transformed to carbide Al_4C_3 is defined as the Quality Factor (QF) and it expresses the measure of the dispersion technology efficiency. For different types of heat treatment it is shown in Table 2. The QF was used in /6/ for empirical correlations of the dependence of strengthening on the dispersed phase content. The correlation was expressed by the following equation:

$$\frac{Rm+500}{1420}(A_5)^{0.219}=QF \qquad (1)$$

where Rm is strength, A_5 is elongation and QF is quality factor.

The maximum value of QF is equal to 1, and this value corresponds to the total transformation of carbon to carbide Al_4C_3.

Table 2

Dispersion technology efficiency, the portion of carbon transformed to Al_4C_3, with heat treatment and quality factor.

Heat treatment: 723 K/ 30 h					Heat treatment: 873 K/ 30 h				
Notation	Commercial mark	% $C \to Al_4C_3$	Vol.% Al_4C_3	QF	Notation	Commercial mark	% $C \to Al_4C_3$	Vol.% Al_4C_3	QF
A	LTD	100	12.6	0.85	A	LTD	100	12.6	1.0
I	KS 2.5	78	9.8	0.75	I	KS 2.5	100	12.6	0.95
C	Spezialschwarz 550	76	9.6	0.82	*C*	Spezialschwarz 550	100	12.6	1.0
B	Spezialschwarz 5	68	8.6	0.86	B	Spezialschwarz 5	100	12.6	1.0
F	Farbruss FW 2	58	7.3	0.87	F	Farbruss FW 2	100	12.6	1.0
G	Flammruss 101	55	6.9	0.81	G	Flammruss 101	98	12.3	0.91
E	Printex 400	50	6.3	0.80	E	Printex 400	94	11.8	0.99
D	Printex 30	36	4.5	0.85	D	Printex 30	92	11.6	1.0
H	Thermax	25	3.2	0.80	H	Thermax	64	8.1	0.92

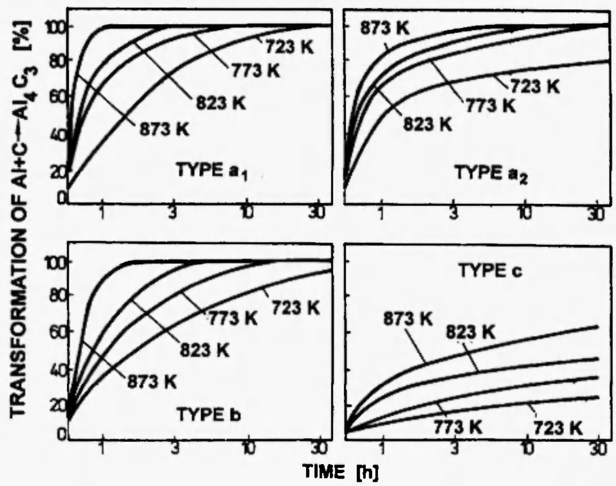

Fig. 1: Dependence of carbon to carbide transformation rate on heat treatment temperature and hold time for 4 carbons types

Microstructure analysis of the produced compacts by optical microscopy showed good 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. Impurities like Al_2O_3 and $FeAl_3$ particles were found in such structure. Residual, quite large carbon particles were observed after heat treatment at 723 K for 30 hours. The distance between the bands was found different. The matrix grain boundaries were not observable.

Analysis by electron microscopy was conducted using carbon replicas and thin foils. It may be noted that the method with carbon replicas was not useful; for quantitative evaluation. Transmission electron microscopy of thin foils offered better results. For all the tested carbon combinations from A to I, thin foils were produced with heat treatment at 723 K for 30 h. The Al_4C_3 particle and subgrain size were measured in 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 not influenced either by the carbon type or heat treatment technology applied.

The mean distance between the particles depended strongly on the carbon type, as it depends on the efficiency of transformation. The 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, resulted from graphite type I (KS 2.5), led to the best production and utilization of this type of dispersion strengthening. It may be added that the results on mechanical behaviour of compacted system described in the next part provide a reason why we do select this material.

Strength (R_m), elongation (A_5) and microstructure of dispersoids are found to depend upon the volume content of the dispersed Al_4C_3 phase as shown in Fig. 2 (723 K/30 h) and these mechanical properties are influenced by the technology applied. Grain size with the nearly constant dispersed particle affects the strength and the plasticity correlates with the subgrain size and the mean dispersed interparticle distance. The effect of low temperature of the heat treatment on carbide reaction (723 K/30 h) revealed more differences in structural parameters, strength and elongation.

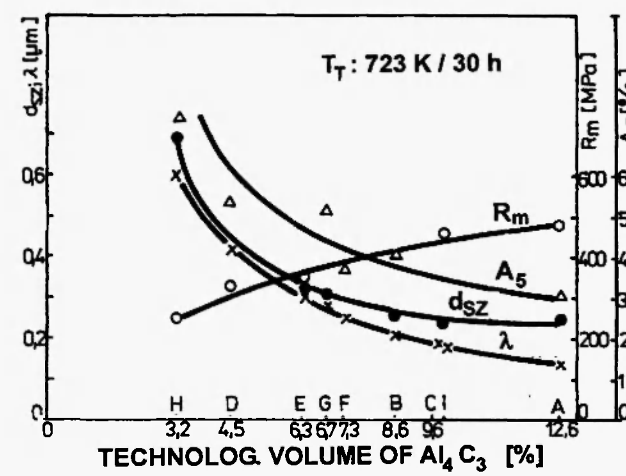

Fig. 2: Mechanical properties and microstructure parameters in dependence on the Al_4C_3 phase content after heat treatment 723 K/30 h

From the viewpoint of strength increase, the carbon types A, C, and I showed the best results. The subgrain size appears to be another measure of microstructure control in the materials presently investigated. The corresponding substructures showing this relation are given in Figs. 3-5.

Low strengthening was measured for materials with carbon of type H, which corresponds to low carbon transformation efficiency and low phase Al_4C_3 volume content. The substructure obtained with carbon of type H is displayed in Fig.6. The effect of the Al_4C_3 phase at

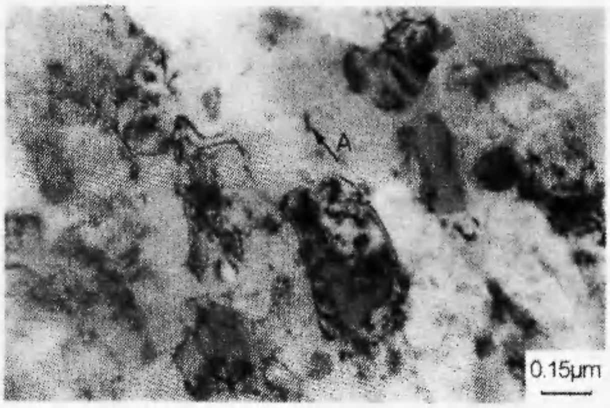

Fig. 3: Substructure of Al-Al_4C_3 system with 3 mass % C of LTD type (A - dispersed phase)

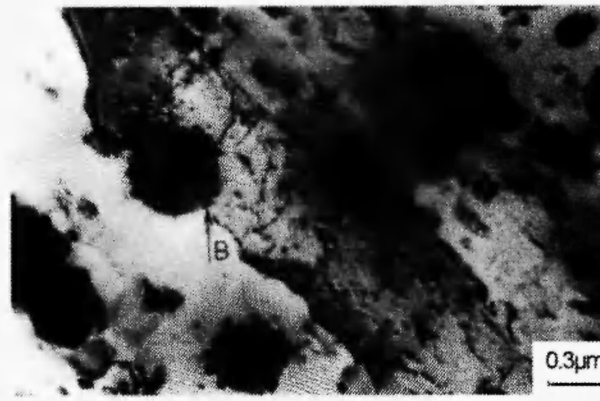

Fig. 6: Substructure of Al-Al_4C_3 system with 3 mass % C of Thermax type (B – graphite)

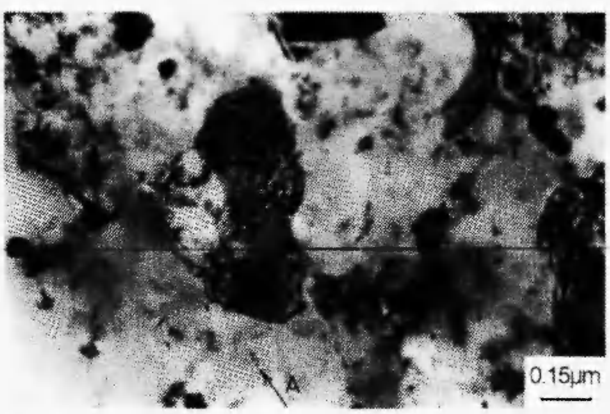

Fig. 4: Substructure of Al-Al_4C_3 system with 3 mass % C of KS 2.5 type (A - dispersed phase)

materials with carbon of types B, F, G, and E was so small that it has not influenced mechanical properties and microstructure parameters. The carbide distribution of these four materials is shown in Figs 7-10. The effect of heat treatment during manufacturing Al-Al_4C_3 materials has been provided in detail in ref. /7. The temperature effect on mechanical properties and fracture of the Al_4C_3 materials has been described in /8/ and discussion is given in the previous work /9/.

Fig. 5: Substructure of Al-Al_4C_3 system with 3 mass % C of Specialschwarz 550 type (A - dispersed phase)

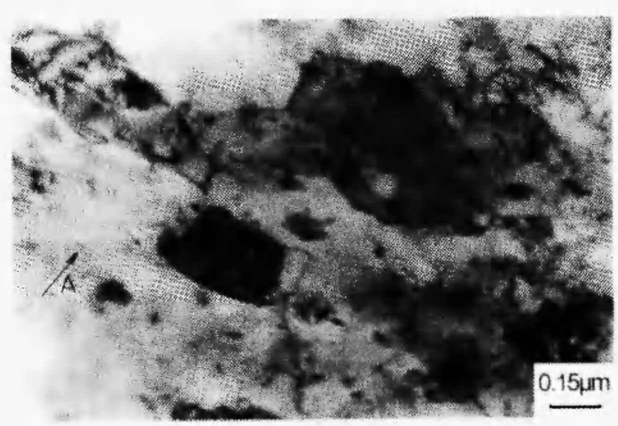

Fig. 7: Substructure of Al-Al_4C_3 system with 3 mass % C of Specialschwarz 5 type (A - dispersed phase)

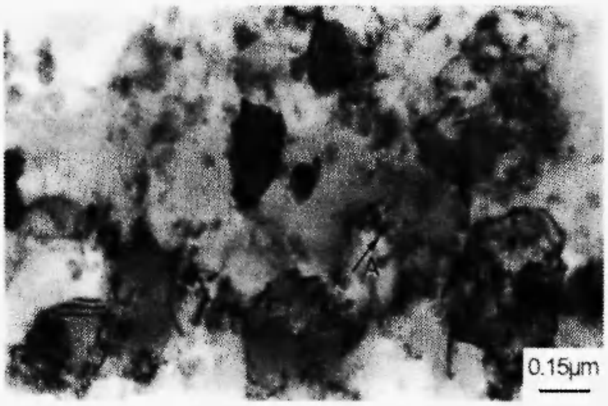

Fig. 8: Substructure of Al-Al_4C_3 system with 3 mass % C of Farbruss FW 2 type (A - dispersed phase)

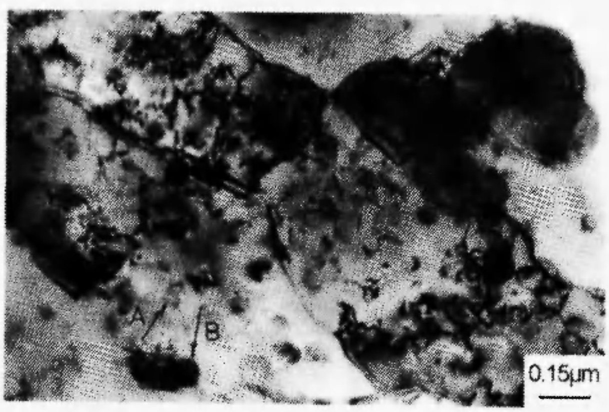

Fig. 9: Substructure of Al-Al_4C_3 system with 3 mass % C of Flammruss 101 type (A - dispersed phase, B - graphite)

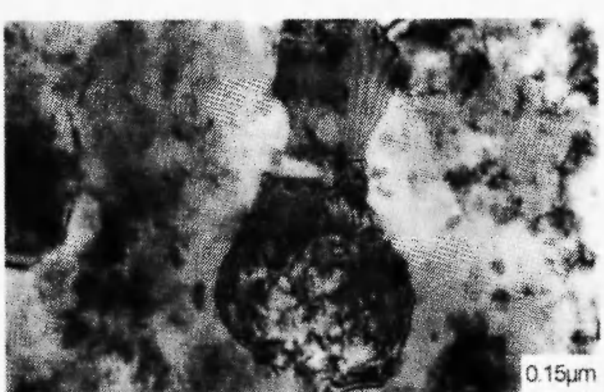

Fig. 10: Substructure of Al-Al_4C_3 system with 3 mass % C of Printex 400 type (A - dispersed phase, B - carbon)

4.CONCLUSION

The present results on mechanical alloying process and heat treatment of Al–C system and on deformation behaviour of dispersion strengthened Al-Al_4C_3 system prepared at different condition can be summarized as follows: It was shown that, in the powder mixture, 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). The volume portion of carbide phase Al_4C_3 and the efficiency of transformation are in good agreement with the microstructure obtained and tested mechanical property. The quality factor *QF* is found to be a good indicator for describing the milling and heat treatment-induced carbon transformation to carbide.

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