THE SUBSTRUCTURE FORMATION AT DISPERSION STRENGTHENED Al-Al_4C_3 MATERIALS

M. Šlesár, G. Jangg

Dedicated to Prof. Jangg at his 75th anniversary

Abstract
The methods of mechanical alloying, carburising heat treatment and compaction by extrusion were used during research and development of dispersion strengthened Al-Al_4C_3 materials. Substructure changes created in the individual stages of treatment are analysed and described in this work. Formation parameters of a thermal high stabile substructure created by dynamic polygonisation up to homology annealing temperature of 0.94 T_m are also analysed.
Keywords: mechanical alloying, dispersion strengthening, extrusion, dislocation substructures, dynamic polygonisation, strength and plastic properties

INTRODUCTION

All production methods of dispersion strengthened materials can be divided into two groups. Namely the methods introducing the hardening phase (oxides, carbides, nitrides) into the matrix, or methods in which such a phase is formed during the production process, most frequently by chemical reaction. In the first case, the parameters of dispersoids and the matrix powder, methods of compactisation or heat treatment, are very important. In the second case, it is necessary to gain the knowledge of the mechanism and of kinetic parameters of dispersion phase formation within the production cycle, to modify the final microstructure and properties of the system.

Powders can be produced by different ways. Their metallurgical purity and its changes resulting from technological operations are of key importance. Surface oxides, contamination of powders, gases and inclusions can lead to the formation and growth of cracks and to the degradation of mechanical properties.

Another factor influencing the structure and final properties is the way of preparation of matrix-dispersoid composition. Gaining the knowledge of the laws of milling, mechanical alloying, salt co-precipitation and internal oxidation is therefore inevitable. Consolidation of composites (pressing, sintering and hot consolidation) to a practically porousless stage is achieved by various deformation processes. Methods of final consolidation at an extremely high degree of plastic flow result in the rearrangement of dispersoids and an increase of accumulated energy of the crystalline structure. The process of hot consolidation can also take place under thermomechanical conditions, then to produce a stable dislocation substructure.

Mechanical properties of dispersion strengthened materials depend on microstructural and substructure parameters and their changes at increased temperatures. The most important are the size, shape, disorientation of grains and subgrains, structure of

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boundaries, dislocation substructure and microstructure heterogeneity. Others include admixtures, imperfect binding of granules at compactisation. All depend on the interaction of the dispersoid with matrix dislocations from the aspect of the mechanism of plastic deformation.

GENESIS AND CHARACTER OF WORK

One of the important themes from the bilateral scientific and technological co-operation between the Institute for Chemical Technologies and Analytics of the Vienna University of Technology and the Institute of Experimental Metallurgy (now Institute of Materials Research) of the Slovak Academy of Sciences in Košice, was the research of alloying, production and properties of materials based on AlC. Among the main themes and goals are:

a) The study of mechanical alloying conditions, granule formation of the Al-C system in laboratory attritor „Netsch“ [1, 2].
b) Analysis of compactisation processes of powder granules by pressing and extrusion [1, 2, 3].
c) Research of microstructure stability of composite materials [4, 5].
d) Research of the strength and plastic properties at room temperature and also at higher ones, as well as a research of the creep properties [6, 7, 8, 9, 10, 11].

The results of analysis of the substructure development in technological production process of the above mentioned materials and their subsequent treatment are described in this work. The density changes, dislocation consolidation, and changes of subgrain size were taken into consideration as the basic substructural parameters.

SUBSTRUCTURE FORMATION OF GRANULES DURING INTENSIVE MILLING

Atomised Al powder, consisting of spherical or ellipsoidal particles, was used as a starting material. The size of particles is characterised by the information that dimensions of 95% of particles were in the range 15-30 μm. Used graphite KS 2.5, was leaf-shaped. The diameter of 90% of the leaves was in the range 1.5-5 μm, and their thickness ranged from 0.1 to 0.5 μm. Intensive milling in a laboratory attritor was carried out during 10-180 min at 900 rpm. Used steel mill balls were 7 mm in diameter. The charge of the AlC1 type consisted of 0.3 kg Al, 3 g C and 6 kg balls. Continual milling was carried out in a tightly closed container of an attritor for determined time periods. In the case of discontinued milling, the mill was opened after determined time intervals of 10, 20, 40, 60, 90, 120 and 180 min, the charge was removed and subjected to sieve analysis. After that, it was returned back to the mill for additional milling necessary to reach the subsequent time limit. Repeated exposure of aluminium to the atmosphere assured that oxidic films developed on the surface of Al particles. Results of sieve analyses were used to plot kinetic diagrams of the development of the granulometric composition of milled material in dependence on time and milling conditions. Figure 1 shows the diagram of conti-milling, and Fig.2 disconti-milling of AlC1. Comparison of these diagrams suggests the existence of quantitative differences the development of granulometry in dependence on the created oxidic films.

X-ray diffraction analyses were used to observe the changes of dislocation density and the size of zones of coherent dispersion during the whole milling process. Figure 3 indicates that the biggest gradient of changes corresponds to the first 10-60 min of the milling process. This is related to the formation of lamellae and the development of first aggregates.

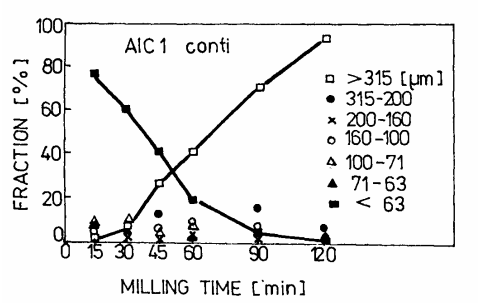

Fig.1. Development of granulometry in dependence on milling conditions.

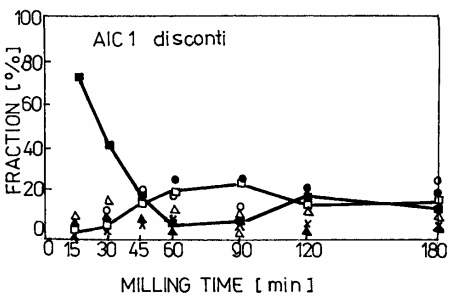

Fig.2. Development of granulometry in dependence on milling conditions.

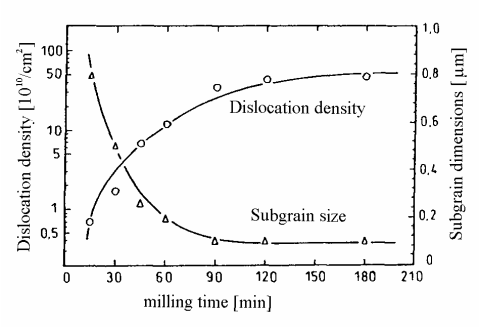

Fig.3. Influence of milling time on substructure parameters.

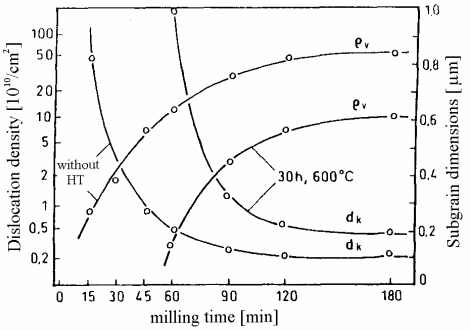

Fig.4. Influence of milling time and heat treatment on substructure parameters.

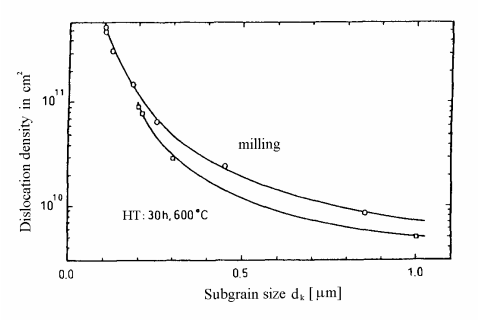

Fig.5. Relation between subgrain size and dislocation density in granules in state after milling and heat treatment.

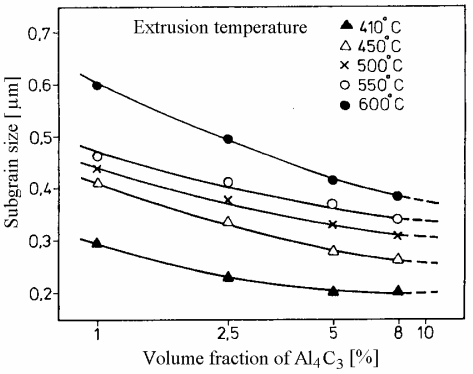

Fig.6. The dependence of subgrain size on volume fraction of Al_4C_3 and on extrusion temperature.

A substantial part of graphite in the form of fine particles is chemically retained during the process of intensive milling. Heat treatment of granules is necessary for the formation of dispersed Al_4C_3 carbides. Heat treatment at 550°C during 5 h or at 600°C

during 1 h, ensure sufficient transformation of graphite particles into carbides when an addition of 1% graphite KS 2.5 is used. This is necessary for attaining good quality of bars compacted by the following extrusion. The changes of substructure formed by milling occurred during heat treatment of granules. An example is shown in Fig.4. The individual curves are the dependencies of dislocation densities and subgrain dimensions in the Al + 1C granules milled for various time periods and after heat treatment for 30 h at 600°C. From this dependence, it follows that a relatively fast formation of dispersed carbides brake the starting recovery processes and grain growth. From the realised experiments, it also follows that there exist relationships between dislocation density and subgrain size in the state after milling as well as heat treatment, Fig.5, [1, 2, 3, 4, 5].

EXTRUSION EFFECT ON GRANULE SUBSTRUCTURE CHANGES

There was also realised research of the effect of AlC volume portion in the range of 1-10%, as well as the effect of extrusion temperature in the range of 450-600°C. Granules were compacted at the pressure of 600 N/mm^2 to the cylindrical form (∅ 24 mm). These specimens were heat treated at 600°C during 1 h with a scope of carburising reactions. Cylindrical specimens after pre-heating to the different temperatures were formed to the bars of ∅ 6 mm by extrusion. The influence of O_2 and Fe for an increase of real volume portion of dispersed particles was taken into account, to appreciate the effect volume portion of Al_4C_3 particles on substructure and yield strength. We had determined that individual specimens contain ca 1.4 mass.% of Al_2O_3 particles after using technology for specimen preparation. They were formed partly from starting Al powders and partly by an increase during milling in attritor. The mixtures contain also fine $FeAl_3$ particles formed from Fe particles released by abrasion from individual mill parts. By this way, ca 0.4% particles of $FeAl_3$ were as formed. These particles contribute by the same degree to the strengthening which was formed by dispersion Al_4C_3 particles.

The testing specimens were prepared from the extruded bars for evaluation of strength and plastic properties and for substructure analysis. The subgrain size was evaluated on thin foils, the dimension and morphology of dispersion particles on carbon replicas by means of electronmicroscopy. The dependence of subgrain size on the volume fraction of Al_4C_3 and on the extrusion temperature is in Fig.6. These dependencies show a high sensitivity of varying technology parameters on the formed substructure. The subgrain size was changed in the range of 0.2–0.6 μm. The size of dispersion Al_4C_3 particles was changed predominantly in dependence on their volume fraction, e.g. for 1% volume Al_4C_3 fraction, the size was 0.010 μm, and 0.035 μm for 10% volume Al_4C_3 fraction.

Identified changes of substructure were significantly expressed also on the strength and plastic values determined by static tensile test. Figures 7 and 8 show the dependence of tensile strength and yield strength for extruded bars on the volume fraction of Al_4C_3 and on extrusion temperature. Plastic properties were characterised by a reduction of area values. The reduction of area at fracture, as a function of the volume fraction of Al_4C_3, and the extrusion temperatures are shown in Fig.9. Analysis of the influence of the individual strengthening constituents is not the aim of this work. In this area there many papers were realised with an accent to the knowledge of the portion for subgrain, dislocation and dispersion particle strengthening effect [6, 7, 8, 9, 11].

Microstructure and substructure composition after extrusion is formed under conditions of dynamic polygonisation. It is very stable also at high annealing temperatures when the presence of dispersion particles is taken into account. This problem is solved also in the next part of the work [8, 9].

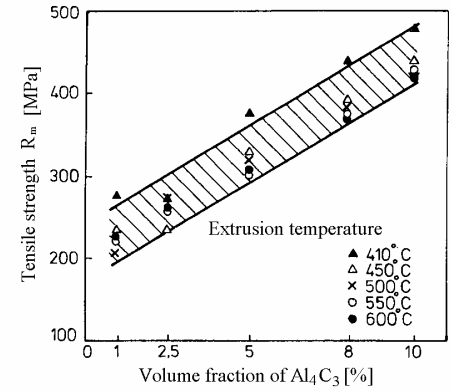

Fig.7. Dependence of tensile strength on volume fraction of Al_4C_3 and on extrusion temperature.

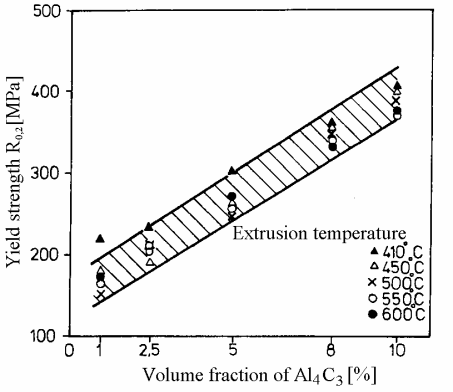

Fig.8. Dependence of yield strength on volume fraction of Al_4C_3 and on extrusion temperature.

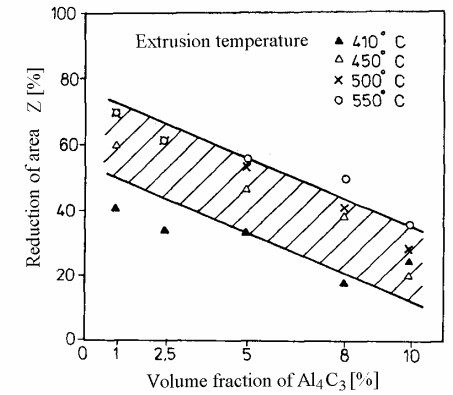

Fig.9. Dependence of reduction of area on volume fraction of Al4C3 and on extrusion temperature.

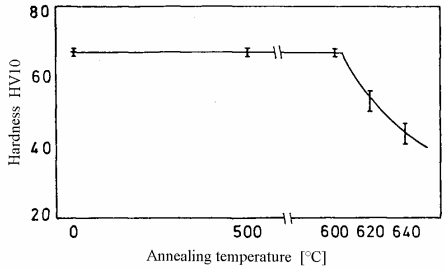

Fig.10. Macrohardness values changes in dependence on annealing temperature for AlC1 material.

SUBSTRUCTURE STABILITY OF DISPERSION STRENGTHENED Al-Al_4C_3 MATERIAL

The task described in this part is very significant because of specification in the knowledge about stability, polygonisation and consolidation processes. Analysed were the influences of :

annealing temperature of dispersion strengthened Al-Al_4C_3 alloys on the changes of hardness and substructure,

alternating of high-temperature softening and deformation strengthening processes on the changes of substructure and hardness.

The effects of annealing temperatures were studied on material with 1 mass.% portion of graphite KS 2.5. The compacts were prepared by extrusion at 580°C. The testing specimens made form bars were annealed at the temperatures of 500, 600, 620 a 640°C during 1 h in an argon atmosphere. The changes of macrohardness HV 10 values are illustrated in Fig.10. The hardness values at given annealing conditions are stable up to a temperature of 600°C. A decrease of hardness values was attained even at 620°C and

640°C. By means of described annealing parameters, the next substructure changes were obtained. The starting material after extrusion at 580°C is in Fig.11. There are visible rectangular subgrains of Al with the mean diameter of 0.8 μm, in subgrains fine Al_4C_3 particles are distributed. By annealing during 1 h at 500°C, a substructure was changed lightly. The mean subgrain size was 1 μm, Fig.12. After annealing at 640°C during 1 h, a substructure characterised in Fig.13 was formed. The processes of subgrain boundary extinction and coarsening of carbide Al_4C_3 particles were in progress. The described changes are the reason of identified changes of macrohardness. High substructure stability remained up to a temperature of 600°C, which corresponds to the homologue temperature $T/T_m = 0.94$.

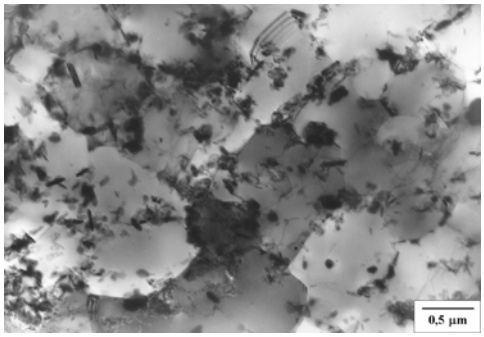

Fig.11. Substructure of AlC1 material after extrusion at 580°C.

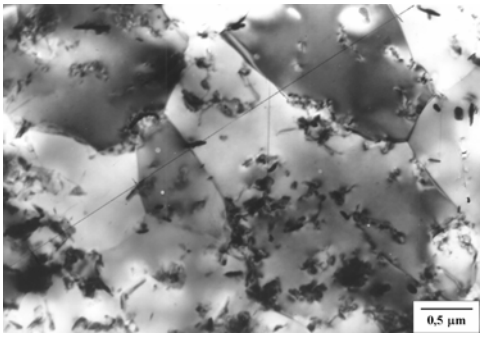

Fig.12. Substructure of extruded AlC1 material after annealing during 1h at 500°C.

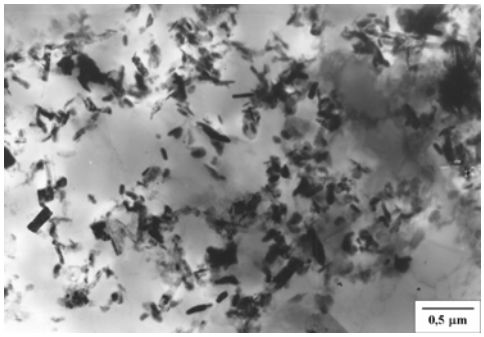

Fig.13. Substructure of extruded AlC1 material after annealing during 1h at 640°C.

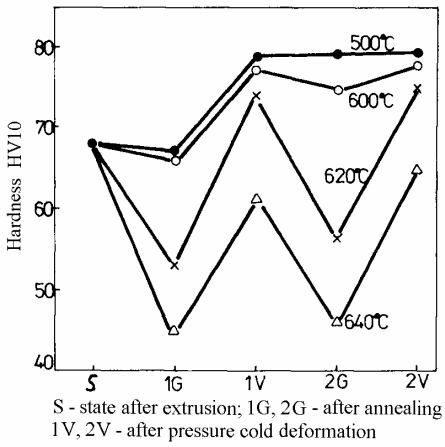

Fig.14. Changes of hardness values after annealing and cold deformation (S – state after extrusion, 1G, 2G – after annealing, 1V, 2V – after pressure cold deformation).

For knowledge about properties and structure stability in the investigated system, the next experiment was carried out – varying of high temperature softening by annealing and the following strengthening by cold deformation. Material AlC1, prepared by extrusion at 550°C, was used. The specimens made from bars were heat treated by annealing at 500,

600, 620 and 640°C in an argon atmosphere. Also in this case, the changes of properties were evaluated by hardness HV 10. Annealed specimens were treated by 50% pressure cold deformation at a deformation speed of $\dot{\varepsilon} \sim 5\text{-}7\ s^{-1}$. For explanation of the hardness changes, there were prepared thin foils for TEM. The results of measured changes of hardness HV 10 are in Fig.14. The starting material as-extruded had a value of HV 10 = 68. The mean size of subgrains at this stage was 0.8 μm. The first annealing, signed 1G, at the temperatures of 500-600°C caused a very small lowering of HV 10 values, annealing at 620-640°C caused a significant decrease of hardness. The specimens, after annealing at different temperatures, were further treated by 50% pressure plastic cold deformation. Substructure refinement occurred in materials annealed at 500-600°C before plastic cold deformation. The main subgrain size was 0.6 μm, and dislocation density increased mainly at subgrain boundaries. The result was an increase of hardness values up to 77 and 79 HV 10, respectively. This stage is signed as 1V in Fig.14.

We noticed that dynamically polygonised substructures were disintegrated after annealing at 620-640°C. The Al_4C_3 particles were coarsened, and they were composed predominantly into disconnected net shapes. After following plastic cold deformation, relatively wide walls of dislocation cells were formed in the areas of carbidic nets. The increase of dislocation density and the dislocation configuration into cells, meant that for the specimens annealed at 620-640°C as well, the hardness HV 10 values increased, but to a lower extent as compared with materials annealed at 500-600°C, Fig.14.

The second annealing at 500-600°C, marked in Fig.14 as 2G, did not cause any substructure changes as well as hardness HV 10. The materials, after plastic cold deformation and annealing at 620-640°C, were significantly softened, which is characterised by a significant hardness decrease. The dislocation cell structure, which was formed by previous plastic cold deformation, was disintegrated at these annealing temperatures. Cycle of deformation strengthening was repeated once more, and attained changes of hardness are in Fig.14 signed as 2V. Very small changes in material substructure were obtained after annealing at 500-600°C. Repeatedly, a significant deformation strengthening effect was attained for materials after annealing at 620-640°C.

The results from the described experiments confirmed a high substructure stability of the Al-Al_4C_3 material after its extrusion and followed annealing up to temperatures of 600°C. On the contrary, the annealing at 620-640°C caused a significant softening. Following plastic deformation in this softened stage will create a deformation strengthening by dislocation density increase, and by formation of dislocation cells. So, a very interesting cycle of substructure stability or hardness changes was identified, and its subsistence was described [4, 5].

CONCLUSIONS

A wide study realised during development of dispersion strengthened Al_4C_3 alloys by powder metallurgy method is summarised in this work. Connections between the technological parameters of mechanical alloying, heat treatment and especially of high temperature extrusion are emphasized. The causality of above-mentioned changes is explained on the basis of detailed analysis of substructure for these materials. Also the relations of substructure to the strength and plastic properties, defined by static tensile test, were evaluated.

The temperature stability of substructures formed by dynamic polygonisation at extrusion and following annealing temperatures is also appreciated in the work. Structural and hardness stability of these materials is well-preserved up to homological temperatures $T/T_m = 0.94$. The reasons of described stability, as well as the

reason of cycle substructure and hardness changes after overloading the limit of annealing temperature are explained.

Inscription

The authors of the paper, during a 20-year collaboration act as leaders of scientific teams. A part of the joint results was summarised by Prof. M. Šlesár as an acknowledgement and reputation for co-operation at the occasion of Prof. Jangg 75th birthday. He had a key portion on the attained results. Hereby we express thanks to all our colleagues who participated in our successful collaboration.

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