QUANTITATIVE MICROSTRUCTURE ANALYSIS OF DISPERSION STRENGTHENED Al-Al_4C_3 MATERIAL BY EBSD TECHNIQUE

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Abstract
The main aim of the present work was described quantitative analysis of microstructure of by electron backscatter diffraction (EBSD) technique of dispersion strengthened Al-Al_4C_3 material. It is a technique by which SEM can be used to evaluate the microstructure by crystallographic analysis based on the acquisition of diffraction patterns from bulk samples. Mechanical properties of dispersion strengthened materials depend on microstructural and substructural parameters, their changes at elevated temperatures and also on grain and subgrain matrix structures. From this point of view the most important microstructural parameters of matrix are size, shape, and misorientation of grains and subgrains and their annealing behaviour. Realized microstructure analyses of Al-Al_4C_3 material were evaluated from two experimental specimens annealed at different temperatures and times. Obtained microstructures were evaluated and compared. Annealing process has influenced the grain size, shape and orientation of experimental material. Grains size and angle grain boundaries were evaluated from acquired crystal orientation maps.

Keywords: EBSD, grain size, angle grain boundaries, misorientation

1 Introduction

Dispersion strengthened aluminium alloys manufactured by mechanical alloying (MA) using powder metallurgy technology are promising structural materials that enable significant weight cut for use first of all in aircraft and automobile industry and also at elevated temperatures [1, 2]. MA produces a homogeneous distribution of inert, fine particles within the matrix and avoids many problems associated with melting and solidification. Raw materials used for MA are pure or alloy powders that have particle sizes from 1 to 200 µm [3]. The powders are mixed and the mixture is milled, usually in a ball mill. The product of milling is then compacted and sintered to obtain bulk material. The particles of strengthening phase can be introduced into the matrix at the beginning of MA process as one of the powders, or they can be formed from pure element powders during the milling, or further manufacturing processes.

The most important factor affecting strength of the material is dispersion of fine particles of stable and hard phase introduced to matrix during MA. These particles serve as obstacles for dislocation motion and anchor subgrain and grain boundaries of matrix [4]. Unfortunately the visualisation of matrix microstructure by light metallography or scanning electron microscopy is extremely difficult and often impossible, due to small grain size or persistency of material to common etchant. Recently, the electron backscattered diffraction (EBSD) technique proved effective tool for quantitative analysis of grain and subgrain structures.

The aim of this paper is to study matrix properties of aluminium strengthened by Al_4C_3 carbide during annealing at higher temperatures by means of EBSD.

EBSD is a quantitative technique that reveals grain size, grain boundary character, grain orientation, texture and phase identity [4]. The basic requirement is a standard SEM, and EBSD system. The EBSD acquisition hardware generally comprises a sensitive CCD camera, and an image processing system for pattern averaging and background subtraction. EBSD is carried out on a specimen which is tilted between 60° and 70° from the horizontal. This is the best achieved by mounting the specimen so that the surface is normal to the electron beam, which is the optimum position for the microstructure examination using backscattered electrons. The diffracted signal is collected on a phosphor screen and viewed with a low-light video camera. These diffraction patterns provide crystallographic information about microstructure of the specimen. As the speed of pattern analysis has increased, it has become practical to scan the beam over multiple points on the sample to create an orientation map (OM). For OM the particular grains (points with identical crystal orientation) are colored according to its crystal orientation using defined color key. Pattern quality depends on surface of the material under examination, individual crystal orientation and strain present in the structure [5 – 11].

2 Experimental materials and methods

The experimental material was prepared by mechanical alloying. Al powder with the maximum particle size of 50 μm was dry milled in an attritor for 90 min with the addition of graphite KS 2.5 powder. Milled powder was then compacted by cold pressing using a load of 600 MPa. Subsequent heat treatment of cylindrical compact at 550°C for 3 hours induced chemical reaction $4Al+3C \rightarrow Al_4C_3$ creating 4 vol. % of Al_4C_3. The cylinders were then hot extruded at the temperature of 600°C with 94% reduction of the cross section. The size of extruded rod was 6 mm diameter and 40 mm length. Due to a high affinity of Al to oxygen the material also contains a small amount of Al_2O_3 particles. The volume fraction of Al_2O_3 phase was estimated to be low, approximately 1-2 vol. %. The rod was cut into specimens 8 x 5 x 3 mm by the electrospark saw for heat treatment. Two different heat treatment were chosen; annealing at 550°C for 1 hour for relieving internal stresses introduced by extrusion and annealing at 640°C for 100 hours for examination of microstructure thermal stability.

Electron backscattered diffraction analysis was carried out on JEOL 6460 scanning electron microscope (SEM) equipped by INCA Crystal EBSD system. Experimental specimens were electrolytic polished prior the EBSD measurements. From acquired diffraction data crystal orientation maps, pattern quality maps and grain boundary misorientation distributions were created.

3 Results and discussion

Understanding of creation EBSD maps is important for micrographs analyses. Signal originates only there, where is intact crystallographic lattice. EBSP pattern is expressly defined by crystallographic lattice parameters. Quality of diffraction patterns depends on several factors, included with parameters like an ideal crystal, preparation of specimens and surface impurities. Diffraction signs were not possible to evaluate in dark localities of the pattern. It is due to very small particles of secondary phase or pores. Quality of diffraction signs is very sensitive to lattice deformation. Invisible shapes (grains, grain boundaries and external defects) are illustrated by pattern quality maps and OM in electron image. The orientation and pattern quality maps of Al-4 % vol. Al_4C_3 material annealed at temperature 550°C for 1 hour are shown in **Fig. 1** and **Fig. 2**. From the OM in Fig. 1 can be seen that microstructure contains relatively large elongated grains, up to 50 µm length and 10 µm thick, and smaller equiaxed grains of the size around 1 µm. The elongated grains are oriented parallely to the extrusion direction, and small grains fill space among them. The careful inspection of PQM in Fig. 2, showing the same area as OM in Fig. 1, leads to conclusion that some elongated grains consist of small equiaxed grains with nearly the same orientation; see upper middle part of both figures. There are some undisolved points, black areas, where the grains are too small to be identified or the pores or distorted crystal lattice exist, in Figs. 1, Fig. 2.

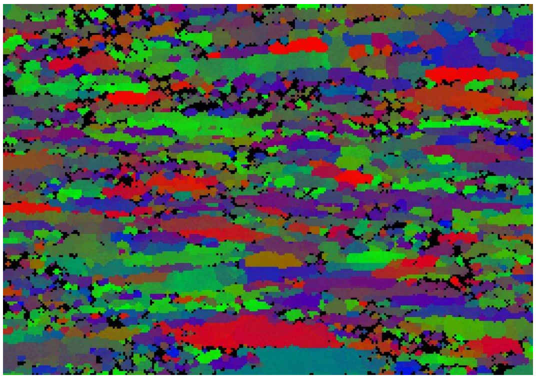

Fig.1 Orientation map of microstructure Al-4 % vol. Al_4C_3 specimen annealed at 550°C/1h

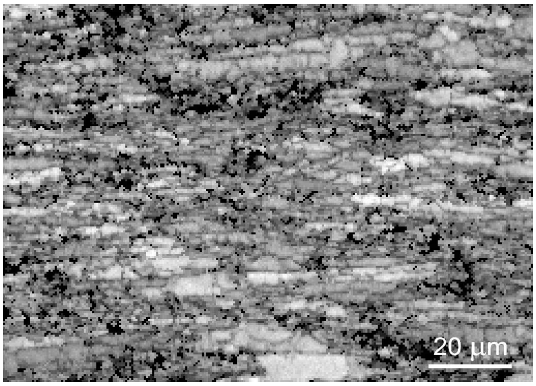

Fig.2 Pattern quality map of microstructure Al-4 % vol. Al_4C_3 specimen annealed at 550°C/1h

The orientation and pattern quality maps of Al-4 % vol. Al_4C_3 material annealed at temperature 640°C for 100 hour are shown in **Fig. 3** and **Fig. 4**. Annealing at higher temperature for longer time leads to substantial microstructure changes. The microstructure is consisted of well developed recrystalized grains of the size between 30 and 100 µm, without any preferential orientation. The crystal lattice in this case is well recovered so it is possible to obtain the EBSD maps of the high quality. The satisfactory agreement between grain boundaries detected in OM and PQM exists. Some additional grain boundaries visible in PQM, Fig. 4, are low angle boundaries which will be mentioned latter. The black dots inside the grains are Al_2O_3 particles.

Typical histograms of grain boundaries misorientation angles of Al-A_4C_3 specimens, which illustrate common classified boundaries with grain misorientation angle less than 15° as low angle grain boundaries (LAGB) and with angle higher than 15° as high angle grain boundaries (HAGB), are shown in **Fig. 5** and **Fig. 6** [12]. The proportion of low angle grain boundaries was determined as 18.4 % for the specimen annealed at 550 °C for 1 hour, and 29.4 % for specimen annealed at 640°C for 100h.

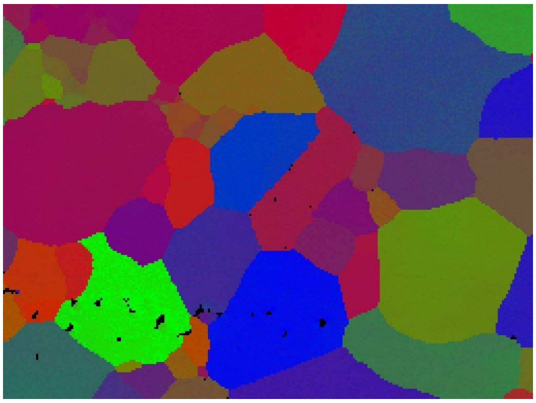

Fig.3 Orientation map of microstructure Al-4 % vol. Al_4C_3 specimen annealed at 640 °C/ 100 h

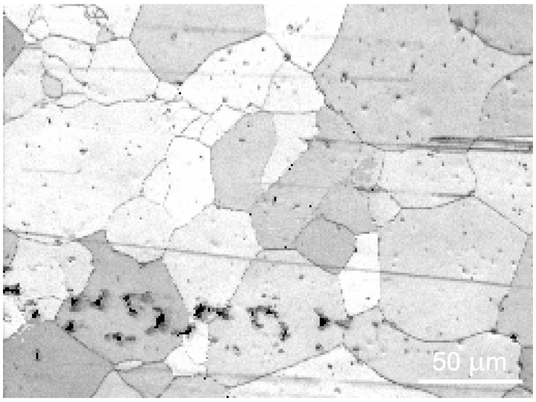

Fig.4 Pattern quality map of microstructure Al-4 % vol. Al_4C_3 specimen annealed at 640 °C/ 100 h

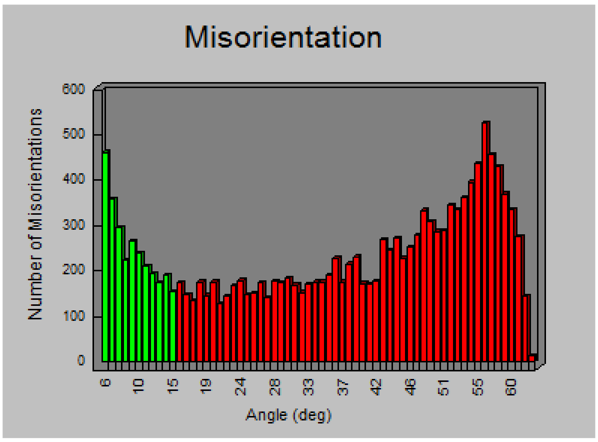

Fig.5 Histogram of grain boundaries misorientation angles for specimens annealed at 550 °C /1h

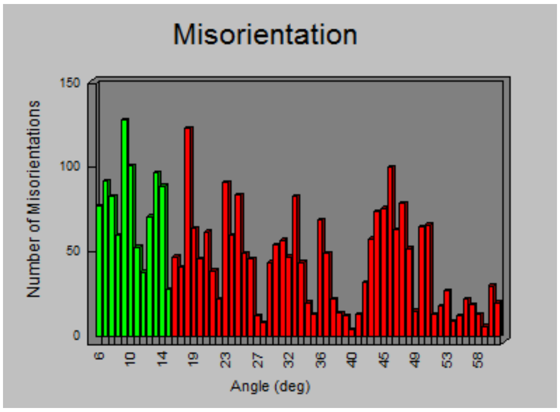

Fig.6 Histogram of grain boundaries misorientation angles for specimens annealed at 640°C/ 100h

4 Conclusions

Microstructures of Al-4 vol.% Al_4C_3 experimental material annealed at two different temperatures and times (550°C/1h and 640°C/100h) were analyzed by electron backscattered diffraction (EBSD) technique. Obtained microstructures were evaluated and compared. Crystal orientation maps, grains size and angle grain boundaries were evaluated. Results demonstrated that quality of maps is very sensitive to lattice deformation. Microstructures illustrate that high angle boundaries are dominated but proportion of low angle grain boundaries was changed. Annealing process has influenced the size, shape and orientation of grains.

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