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MICROSTRUCTURAL EVOLUTION IN MECHANICALLY ALLOYED AI BASED COMPOSITES

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Abstract

The microstructural evolution of mechanically alloyed (MA) AlCu4.5 and AlCu 4.5 Mg1.5 alloys was studied in the course of energetic milling, as well as consolidation process. The MA alloys have nanocrystalline features with extremely deformed fine grain structure. This character assures the excellent mechanical properties at ambient and high temperature.

Introduction

The term "mechanical alloy" means that state which prevails in a composite metal particle produced by agitation milling with high energy. The plurality of constituents in the composite powder can be reached by the application of mechanical energy in the form of repeated welding, fracturing and rewelding of powder particles. The "high energy milling" deforms the deformable metal(s) and causes it to bond or weld to itself and/or to the remaining constituents. The structure of the resulting composite metal particles becomes very fine and homogeneous [1].

A proper quantity of process control agents (PCA) is necessary to add to the metal powder mixture to moderate the strong welding tendency of aluminium and the other elements as well as to prevent the welding between the composite powder and the wall of the mill chamber, the surface of grinding balls and impellers.

At the completion of the mechanical alloying process the powder particles have an extremely deformed metastable structure containing supersaturated solid solution content, zones, clusters, metastable nanocrystalline phases and dispersoids. Inside the particles the homogeneity range is much less than one micrometer.

The most mechanically alloyed powders are generally consolidated by cold or hot compaction (pressing) followed by degassing, sintering and/or hot extrusion, or by hot isostatic pressing.

Objective was to investigate the changes of microstructure of MA powder in progress of the mechanical alloying and to study the variation of the microstructure of the composite material in the course of the consolidating processes carried out by powder metallurgical (PM) route.

Experimental

Al-Cu4.5% and Al-Cu4.5%-Mg1.5% alloys were prepared by mechanical alloying. Elemental aluminium and alloying elements (Cu, Mg) with particle size of 40 μm and less than 4 μm , respectively were blended and put into an attritor with 5 l volume capacity. Stearic acid as PCA was used in amount of 1.5-2 %, the rotating speed of impeller was 200-300 rpm, and the full milling time was 16-32 hours depending on the alloy system.

Monolithic composite was fabricated by cold compactoin (500 MPa), degassing (at 500 $^{\circ}\text{C}$ for 1 hour) and hot extrusion (at 380-400 $^{\circ}\text{C}$, 400- 450 MPa) to reduction of area of 35:1.

The progress of the mechanically alloying as a function of milling time was monitored by means of X-ray diffraction (XRD) method, microscopic investigations (SEM, EPMA, TEM) as well as particle size- and microhardness measurements of powder particles. The microhardness measurements were made on powders with particle size 10-90 μm .

The characterization of microstructures was carried out by the determination of grain size, solid solution content, distribution of alloying elements, identification of intermetallic compounds formed during different technological steps. The microstructural investigations were extended by ambient and high temperature mechanical tests in the case of MA-AlCu4.5Mg1.5 composite-endproducts.

Results and discussion

The microstructural variation and the distribution of the alloying elements of the individual powder particles were followed during the alloying process. In the ternary alloy, at the early stages of the process the metal powders were still rather soft and the tendency for them to weld into large particles predominated. After four hours a broad range of paricle sizes (1-100 μm) developed (Figure 1). Here the particles were layered composites of the starting constituents (Figure 2a). The chemical composition of the composite particles varied significantly within the particles and from particles to particles.

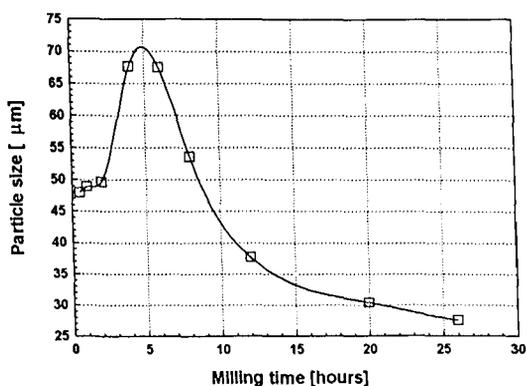


Figure 1. Change of particle size as a function of milling time

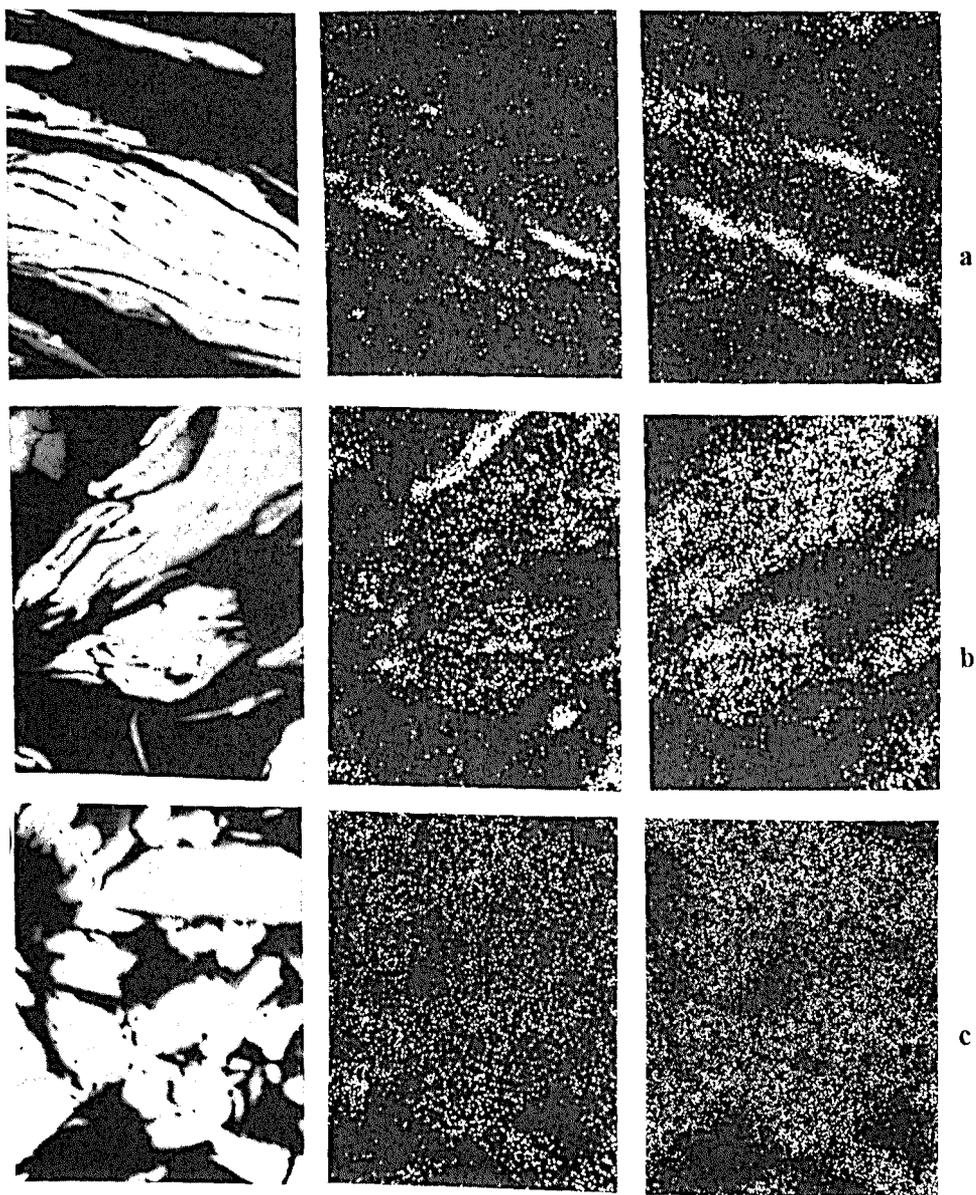


Figure 2. Microstructural variation of powder particles as a function of milling time, BEI, CuK_α , Mg K_α (a: 4 hours, b: 12 hours, c: 26 hours)

As the process proceeds, after 12 hours, the particles got harder, and their ability to withstand deformation without fracturing decreased. The composite powder particles were further refined as fracturing and cold welding continued. In this intermediate stage of processing the particles consisted of convoluted lamellae (Figure 2b). In the intimate mixture of the powder constituents the diffusion distances decreased to the micrometer range. Appearance of poorly crystalline intermetallic phases was observed by XRD investigation in the powder particles.

Near to the end of the process, after 26 hours, the larger particles were broken due to the continuous strike of the steel balls. Then the tendency to weld and to fracture came into balance, and the size of the particles became constant within a narrow range. Then the structure of the composite powder particles became fine layered, convoluted and steadily refined (Figure 2c). The composition of individual particles converged toward the overall composition of the starting powder blend. The lamellar spacing was one micrometer or less and approached the optimum dispersoid spacings.

At this time the microhardness of the individual powder particles attained the saturation level [2, 3]. Saturation microhardness of the MA particles in the binary alloys was about 160, however this value reached 240 in the ternary system (Figure 3).

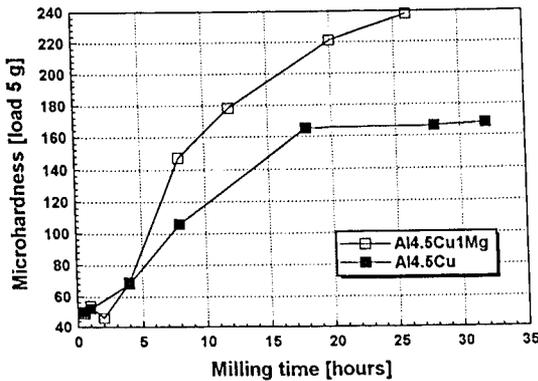


Figure 3. Change of microhardness as a function of milling time

The change of the lattice parameter of aluminium shows the dissolution of Cu and Mg in the course of alloying process (Figure 4). In the binary alloys the dissolution of copper began after 8 hours. The decreasing lattice parameter of aluminium means an isomorphous substitution of copper because its atomic radius is less than that of aluminium. Supposing the same substitution mechanism of copper in the ternary system it can be seen that the dissolution of magnesium began earlier, after two hours. It is shown by the increasing lattice parameters. (The atomic radius of magnesium greater than that of aluminium). After 8 hours the isomorphous substitution of aluminium by magnesium stopped and the dissolution of copper was merely continued.

After milling for 26 hours about 40 % of the copper and 50 % of the magnesium content were in solid solution. The rest of alloying content appeared as poorly crystallized S - (and θ) phases as well as in elemental copper form with coherent particle size of less than 50 nm.

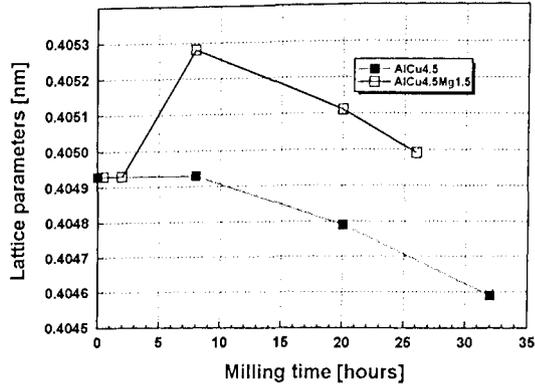


Figure 4. Change of Al lattice parameter as a function of milling time

The mechanically alloyed AlCu4.5Mg1.5 powder showed nano- structural behaviour in overwhelming part (Figure 5). The particle size of the matrix was found to be less than 50 nm and a consequence of it the electron diffraction pattern of aluminium shows continuous rings [3].

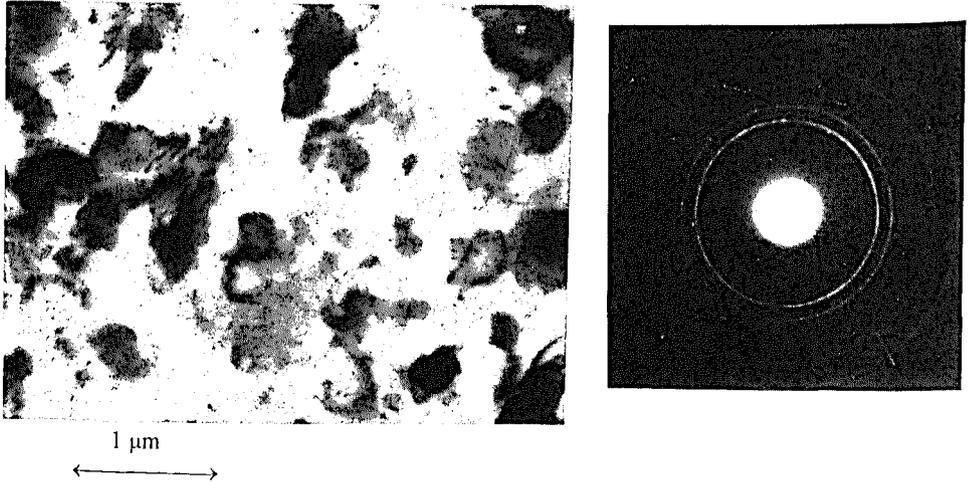


Figure 5. Nanocrystalline structure of MA powder (26 hours), ED pattern

The microstructure of the extruded bars manufactured from the MA powder saved partly the nano-structural features (Figure 6). In the monolithic bulk material besides the Al_4C_3 and Al_2O_3 particles with 20-50 nm particle size formed "in situ" under thermomechanical treatments (hot extrusion) secondary Al_2Cu and S - AlCuMg compounds also precipitated (Figure 7). However a noticeable amount of precipitation of copper and copper - rich particles (Al_2Cu) with about 1 μm size could be observed in the extruded rods (Figure 8).

Both the naturally and artificially aged (T6) extruded bars from MA-AlCu4.5Mg1.5 alloy powder have rather good mechanical properties at ambient and elevated temperature (Figure 9). As comparison the same mechanical parameters of the AA2024 alloy are also presented [4].

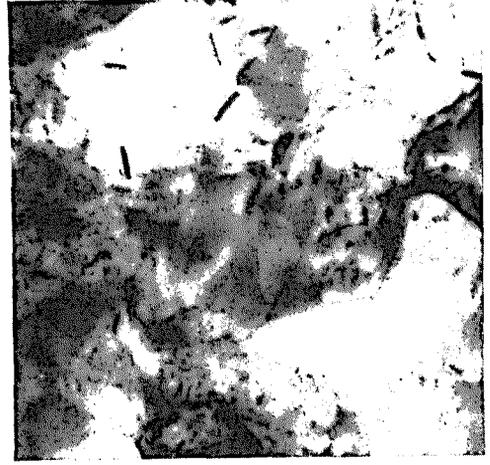
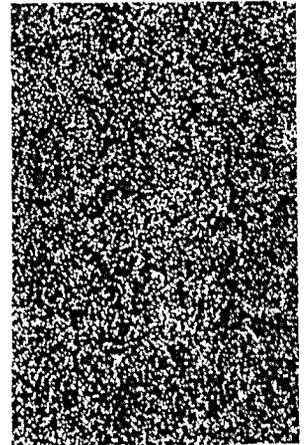
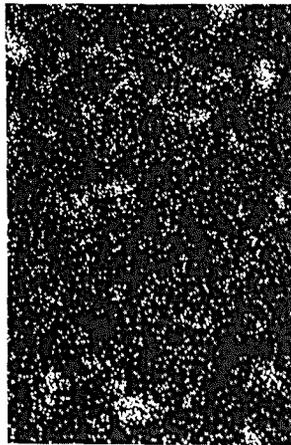
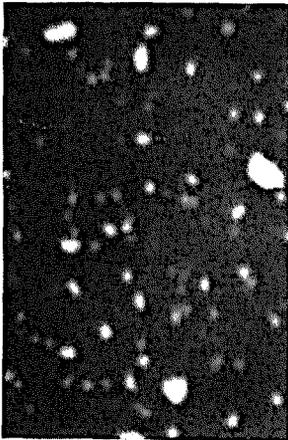


Figure 6. Nanocrystalline structure of as extruded MA-AlCu4.5Mg1.5

Figure 7. Oxide, carbide particles and Al₂Cu dispersoids formed during hot extrusion



10 μm



BEI

CuK_α

MgK_α

Figure 8. Copper and copper - rich particles formed in the course of hot extrusion

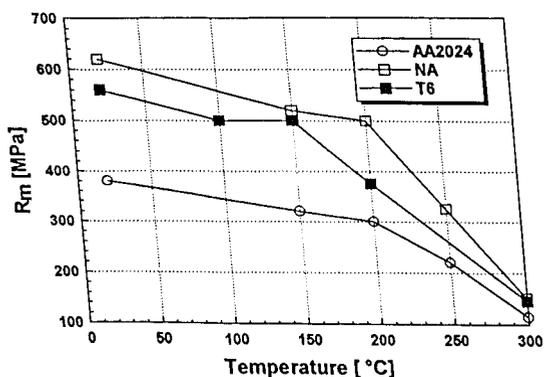


Figure 9. Ultimate tensile strength of MA-AlCu4.5Mg1.5 extruded material as a function of temperature

Conclusion

1. The MA AlCu4.5Mg1.5 alloy powder has a very complex and fine grains structure. This microstructure could be characterized by nanocrystalline state.
2. The MA powder possesses relatively high solid solution content (Cu and Mg).
3. The (mainly) nano- crystalline structure giving the great homogeneity of alloying elements and the grain structure, the high solid solution content of copper and magnesium in the aluminium matrix, as well as the extreme strongly deformed structure containing intermetallic- and/or oxide-carbide- dispersoids almost totally remain after the consolidating process in the bulk material.
4. The good thermostability and the relatively high mechanical parameters of the mechanically alloyed AlCu4.5Mg1.5 alloy can be attributed to its very homogeneous and special structure. These structural features assure the very good strength properties and the thermostability of the MA/PM alloys (or composites) up to 300 °C.

Acknowledgment

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