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# Aluminium Based Alloys with High Content of Metastable Phases Prepared by Powder Metallurgy

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In this paper, the results of preparation and mechanical testing of AlFe7Mn4 alloy are presented. The alloy was prepared by rapid solidification (melt spinning) in the form of thin ribbons. The ribbons were compacted by spark plasma sintering in three different ways — whole, cut into stripes, and crushed into powder in a ball mill. Microstructures of compact materials were observed by scanning and transmission electron microscope. Phase composition was characterized using X-ray diffraction. Rapidly solidified ribbons contained a quasicrystalline phase, but it was transformed to stable phase during compaction independently on the ribbons pre-treatment. Bulk material prepared from whole ribbons exhibited anisotropy of microstructure and mechanical properties. Material prepared from cut stripes showed also slight preferential orientation of ribbon pieces. Crushing of ribbons in a ball mill led to contamination of the alloy powder and to deformation strengthening of bulk material.

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## 1. Introduction

Aluminium alloys have wide applications in automotive and aerospace industries due to their low density. During the last few decades, aluminium alloys prepared by non-conventional techniques like rapid solidification or mechanical alloying were introduced. These alloys have much higher yield and ultimate tensile strength than conventional aluminium alloys and also exhibit higher thermal resistance. They are usually prepared by 2-step powder metallurgy. In the first step, the alloy with intended composition is prepared in the form of powder or thin ribbons that are subsequently milled into powder. Due to rapid quenching of the melt or extreme plastic deformation during mechanical alloying, the alloys are in non-equilibrium state, which means that they are containing supersaturated solution of alloying elements in matrix and also other metastable phases e.g. quasicrystals. The following step of powder metallurgy is compaction of the powder. The compaction conditions should preserve the remarkable properties of powder alloys. As aluminium and its alloys are covered by passivation oxide layer in atmosphere, only few compaction techniques are suitable and among them spark plasma sintering seems to be the best one due to short high-temperature exposure during sintering.

It has been proven that phase transformation of metastable quasicrystals decomposition is accompanied by the change of their shape from spherical to rosetta-like [1]. This effect can be possibly used to close initial states of cracks and so to give to the material so-

called self-healing properties. To prove this theory, it is necessary to prepare bulk material with content of quasicrystalline phase. It has been proven that in Al-Fe-Mn system, quasicrystalline phase with appropriate composition can be thermodynamically stable upon formation [2]. In majority of cases, the quasicrystals are formed during rapid solidification and decompose during subsequent heat treatment [2, 3]. The exact temperature of decomposition strongly depends on chemical composition of the quasicrystal. Slightly alloyed quasicrystals are detectable after annealing at 390 °C [2] and even after compaction by hot pressing at 550 °C [3]. On the other hand, material composed purely of Al, Fe, and Mn prepared by rapid solidification and hot forging at 550 °C contained only stable phases.

The aim of this paper is to compact rapidly solidified AlFe7Mn4 ribbons by spark plasma sintering technique and to describe the influence of ribbons pre-treatment on structure and properties of prepared material.

## 2. Experimental

Master alloy with compositions of Al-7 wt% Fe-4 wt%Mn was prepared by melting of appropriate amount of pure metals in induction furnace followed by melt spinning process with circumferential speed of cooling wheel of 40 m/s. The rapidly solidified ribbon was 20–30  $\mu\text{m}$  thick, 6 mm in width and has length of several meters. Microstructure of rapidly solidified ribbons was studied by transmission electron microscope JEOL JEM 3010 operated at 300 kV (LaB<sub>6</sub> cathode, point resolution = 1.7 Å). Subsequently the ribbons were compacted by spark plasma sintering technique (SPS, FCT System HP D-10) at 500 °C for 15 min. A part of ribbons was crushed in a ball mill (Retsch PM 100, steel vessel) at 400 rpm for 1 h. Microstructure of compact materials was observed by scanning electron microscope (SEM,

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TESCAN VEGA 3, LMU) and transmission electron microscope (TEM, JEOL 2200FS). The TEM samples were prepared by ion polishing using Gatan PIPs. Phase composition was determined by X-ray diffraction (PAN analytical X'Pert PRO + High Score Plus, Cu anode). Compression tests were performed on 3 mm cubes using universal testing machine LabTest 5.250SP1-VM.

### 3. Results and discussion

Rapidly solidified AlFe7Mn4 ribbons had very fine microstructure and were formed by grains of fcc-Al matrix (bright areas) and quasicrystalline phase Al<sub>86</sub>(Mn,Fe)<sub>14</sub> (dark areas), as illustrated in Fig. 1. After consolidation of ribbons, fcc-Al matrix grains coarsening can be observed, as documented in Figs. 2 and 3. Material shown in Fig. 2 was consolidated from ribbons arranged layer by layer perpendicular to the direction of uniaxial pressing during sintering. This led to high anisotropy in properties, which shows the comparison of compression curves in Fig. 4. For compression-testing direction similar to uniaxial-pressing direction by sintering, the material exhibited yield strength of 368 MPa and values for ultimate compression strength and elongation could not be determined because of materials high ductility. On the other hand, sample tested in compression direction perpendicular to uniaxial pressing direction by sintering exhibited yield strength of 318 MPa, ultimate compression strength of 355 MPa and elongation of 8.9%. This can be explained by presence of oriented oxide layers that make the material brittle.

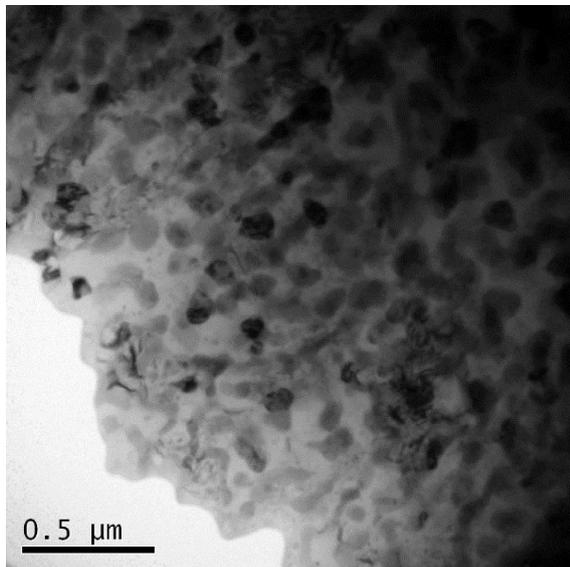


Fig. 1. Microstructure of rapidly solidified AlFe7Mn4 ribbon (TEM).

Two different approaches were tested to suppress the preferential orientation of ribbons used for compaction. The first one was cutting of rapidly solidified AlFe7Mn4 ribbons to the smallest possible pieces by scissors. It resulted in stripes with length about 1 mm. These stripes

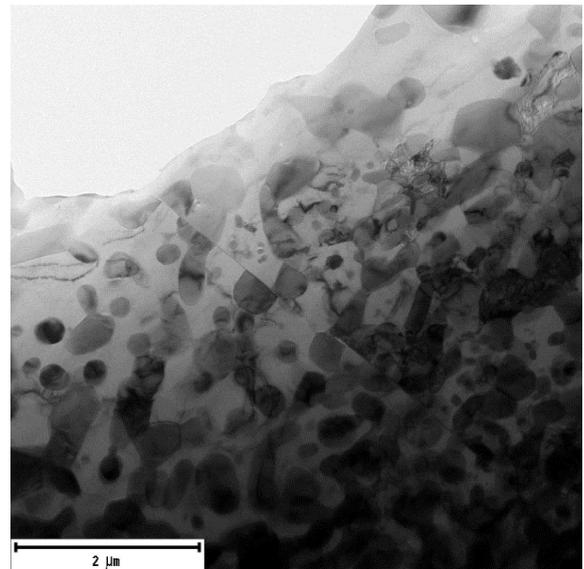


Fig. 2. Microstructure of bulk AlFe7Mn4 consolidated from whole ribbons (TEM).

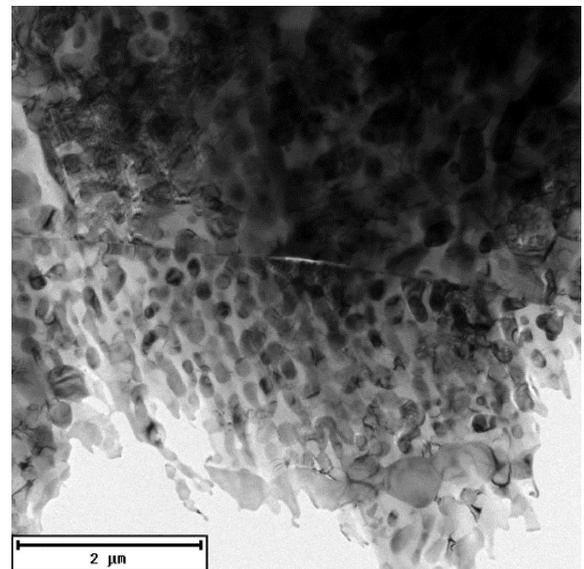


Fig. 3. Compression curves of bulk AlFe7Mn4 samples.

were used for compaction with expectation to obtain randomly oriented microstructural artefacts. In detailed view by TEM, the materials prepared by compaction of whole ribbons and cut stripes of ribbons look similar, see Figs. 2 and 3. The SEM observation in Fig. 5 proved that the orientation of compacted stripes is not completely random. The compression curve of material sintered from stripes is comparable with the compression curve from material sintered from whole ribbons and tested in parallel direction.

To obtain powder as initial material for sintering, the rapidly solidified ribbons were crushed in a ball mill for 1 h. The microstructure of material sintered from the powder is shown in Fig. 6. Severe plastic defor-

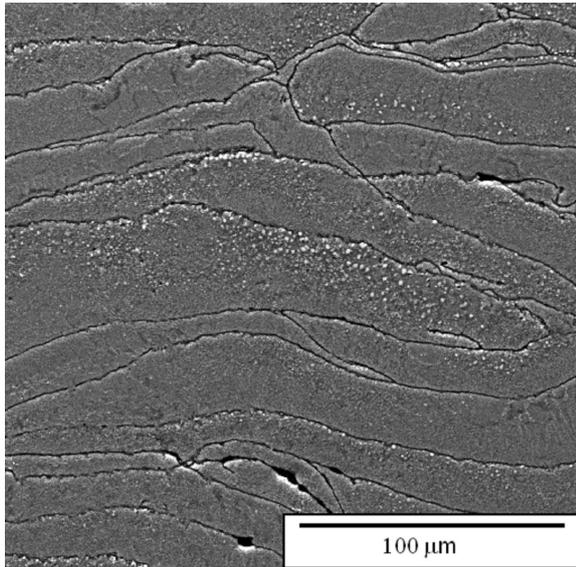


Fig. 4. Microstructure of bulk AlFe7Mn4 consolidated from randomly oriented stripes from ribbons (TEM).

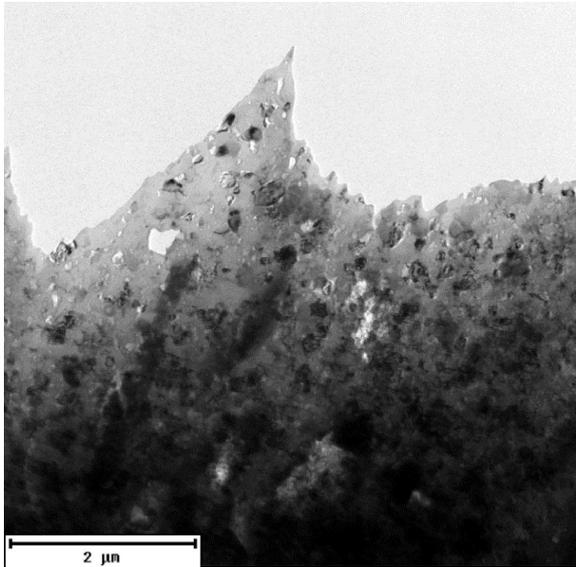


Fig. 5. Microstructure of bulk AlFe7Mn4 consolidated from randomly oriented stripes from ribbons (SEM).

mation during milling has completely changed the microstructure — the spherical quasicrystals were decomposed and the polyhedral heterogeneities visible in Fig. 6 are impurities from milling (SiC). The compression curve in Fig. 4 exhibits signs of deformation strengthening of this material. The changes are also visible by comparing XRD patterns in Fig. 7. Rapidly solidified ribbons contained fcc-Al and quasicrystalline  $\text{Al}_{86}(\text{Mn,Fe})_{14}$  phase. During the sintering process, the quasicrystalline phase decomposed and stable  $\text{Al}_6(\text{Fe,Mn})$  phase was formed. The phase composition after compression testing was the same, which is caused by disappearing of metastable phases already by sintering. The sample prepared from milled ribbons had different phase composition. It con-

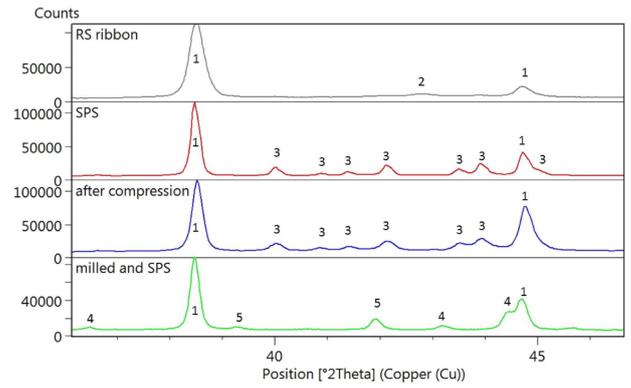


Fig. 6. Microstructure of bulk AlFe7Mn4 consolidated from milled ribbons (SEM).

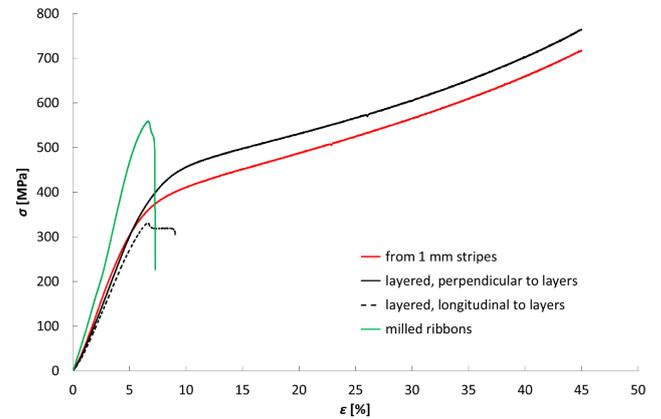


Fig. 7. XRD patterns, 1 — fcc-Al, 2 — quasicrystalline  $\text{Al}_{86}(\text{Mn,Fe})_{14}$ , 3 —  $\text{Al}_6(\text{Mn,Fe})$ , 4 — SiC, 5 —  $\text{SiO}_2$ .

tained fcc-Al but the other minor phases came mainly as contamination from a milling vessel, which is the crucial problem for ball milling process. To protect the microstructure and prevent contamination, a vessel form different material should be used accompanied by slower milling and if possible also cooling by e.g. ethanol. With this approach it may be possible to use milder sintering conditions, because the oxide layer will not be continuously surrounding each sintered particle. Milder condition can preserve quasicrystalline phase during sintering and so brings interesting properties to the material.

#### 4. Conclusion

It was shown that the rapidly solidified ribbons of AlFe7Mn4 alloy can be successfully sintered by spark plasma sintering at 500 °C with process duration of 15 min. This process unfortunately led to decomposition of quasicrystalline  $\text{Al}_{86}(\text{Mn,Fe})_{14}$  phase contained in the initial ribbons that was supposed to give the compact material the possibility of self-healing properties. Material prepared by sintering of whole ribbons exhibited anisotropy in properties. It was slightly improved by

pre-treatment of the ribbons by cutting. Pre-treatment of ribbons by ball milling was not successful because of change in microstructure and contamination of material.

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