

The Influence of Rapidly Solidified Ribbons Pre-treatment on Structure of Bulk AlFeMm Alloys Prepared by Powder Metallurgy

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Rapidly solidified AlFe7Mm4 ribbons were prepared by melt spinning process. The ribbons were composed from super saturated solid solution of alloying elements in Al matrix, stable intermetallic phases and metastable quasicrystalline phases. Both metastable phases can potentially provide self-healing properties to the material – super saturated solid solution by precipitation and quasicrystals by decomposition to stable phases. Key problem of processing such materials is to preserve the microstructure of rapidly solidified material during its compaction. Spark plasma sintering enables powder solidification in very short time – in range of few minutes. This article describes the best way of rapidly solidified ribbons before SPS compaction to obtain bulk material. The ribbons were solidified in the initial state and after cryo-milling. Ball-milling before of rapidly solidified ribbons was also tested. Vickers hardness HV0.01 was also evaluated. Cryo-milling was chosen as an optimal pre-treatment before compaction by SPS.

Keywords: Al alloys, self-healing, SPS consolidation

1 Introduction

Rapidly solidified aluminium alloys with transition metals (TM) are known for their excellent mechanical properties and thermal stability [1-3]. Addition of Mischmetal (Mm, mixture of Ce, La, Nd, Pr) increases the ability of system to form metastable phases (mainly the amorphous metallic glass) [1]. It was also proven that the Mm influence is higher than influence of pure Ce [2].

The Al-TM-Mm alloys are composed of super saturated solid solution (SSSS) of alloying elements in Al matrix, stable intermetallic phases and metastable quasicrystalline phases. Metastable phases can potentially provide self-healing properties to the material, which means the ability of closing and healing a crack formed in the material [4]. The SSSS can behave similarly to underaged alloy. For commercial aluminium underaged (T3) alloy AA2024 was already proven the self-healing behavior [5]. For industrial application, the rapidly solidified ribbons have to be processed by powder metallurgy into bulk materials. The best compaction method is spark plasma sintering (SPS) due to short sintering times [6,7]. This work presents the influence of pre-treatment of ribbons (in form of milling) on properties of prepared powder and bulk alloy.

2 Experimental

The AlFe7Mm4 (composition given in wt.%) alloy was prepared by melt spinning process with circumferential speed of cooling wheel of 40 m/s, which resulted in ribbons with thickness approximately 20 μm . The ribbon were sintered in as-prepared state, after ball-milling (ball mill (Retsch PM 100) at 400 rpm for 1 h) and after cryo-milling (Sencor SCG 1050BK, ribbons milled in ice for 5 min). The ribbons and cryo-milled powder were compacted by Spark Plasma Sintering technique (SPS, FCT Systeme HP D-10) at 500°C for 15 min with pressure of 43 MPa for samples B1 and B3. Sample B2 was compacted using SPS 10-4 Thermal Technology LLC at 450°C for 5

min with pressure of 80 MPa. Sample B3 was pre-pressed before sintering using LabTest 5.250SP1-VM with pressure of 350 MPa.

Phase composition was determined by X-ray diffraction (XRD PANalytical X'Pert Pro). Vickers hardness HV0.1 was measured.

Microstructure of materials was observed by optical microscope (LM, Olympus PME3) and transmission electron microscope (TEM, Jeol 2200FS). The TEM sample was prepared by ion polishing using Gatan PIPs.

3 Results and Discussion

Structure of as-cast AlFe7Mm4 alloy is shown in Fig. 1. It is composed of Al matrix, large needles of $\text{Al}_{13}\text{Fe}_4$ phase and smaller particles of $\text{Al}_{11}\text{Ce}_3$ phase. The phase composition was proved by XRD, given in Fig. 2.

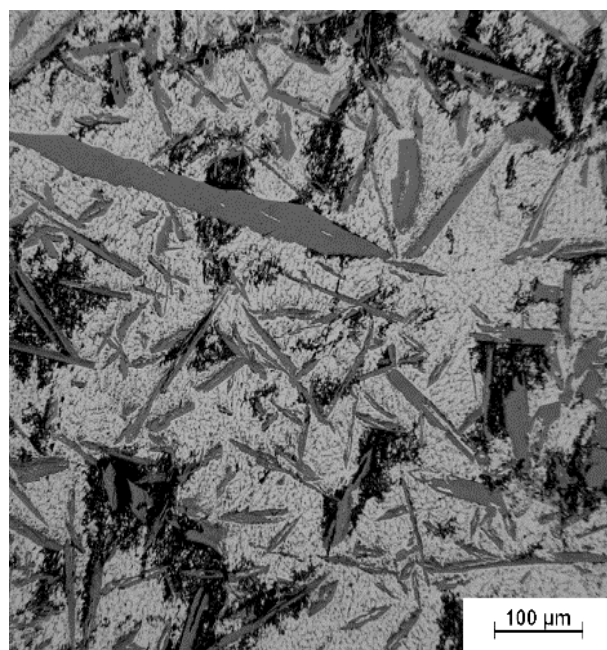


Fig. 1 Microstructure of as-cast AlFe7Mm4 alloy(LM)

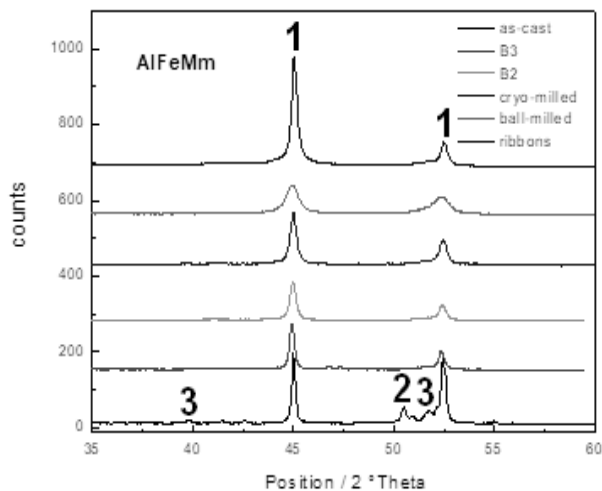


Fig. 2 XRD patterns of different processing of AlFe7Mm4 alloy

After processing by melt spinning, the microstructure was very fine, as illustrated in Fig. 3.

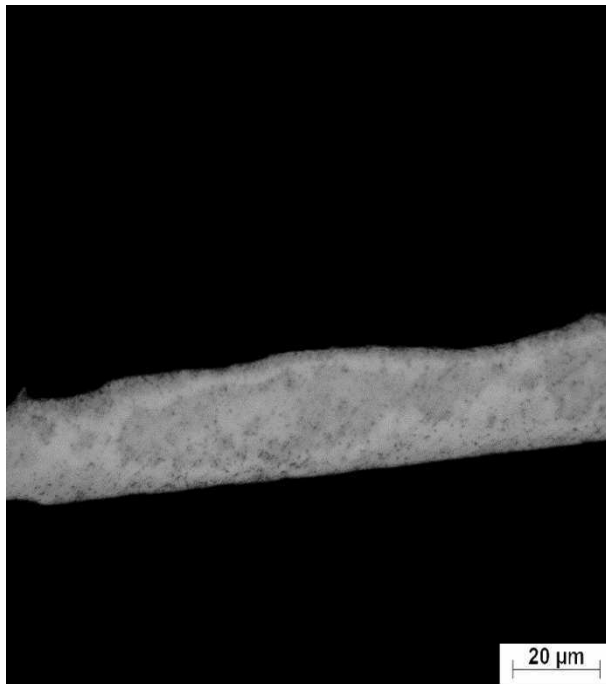


Fig. 3 Microstructure of rapidly solidified AlFe7Mm4 ribbon (LM)

Detail observation by TEM has shown presence of fine grains (light parts in Fig. 4), Al₁₃Fe₄ (dark needles) and quasicrystalline phase Al₈₆Fe₁₄ (dark round phase). Mm was dissolved in the matrix. All intermetallic phases are too fine to be detected by XRD in Fig. 2.

The microstructure of ball-milled ribbons is shown in Fig. 5. The material is dramatically changed compared to rapidly solidified ribbons. The grain refinement was also proven by XRD peak broadening in Fig. 2. Because of these changes in material, this powder was not used for following compaction.

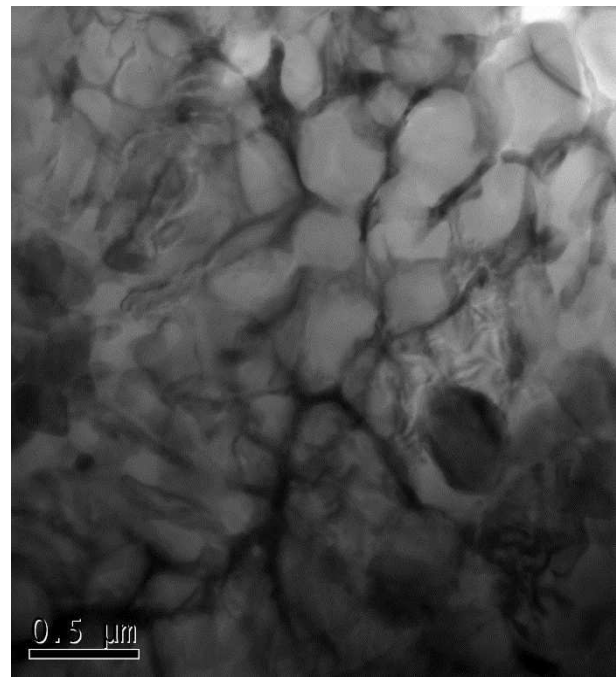


Fig. 4 Detail of microstructure of rapidly solidified AlFe7Mm4 ribbon (TEM)

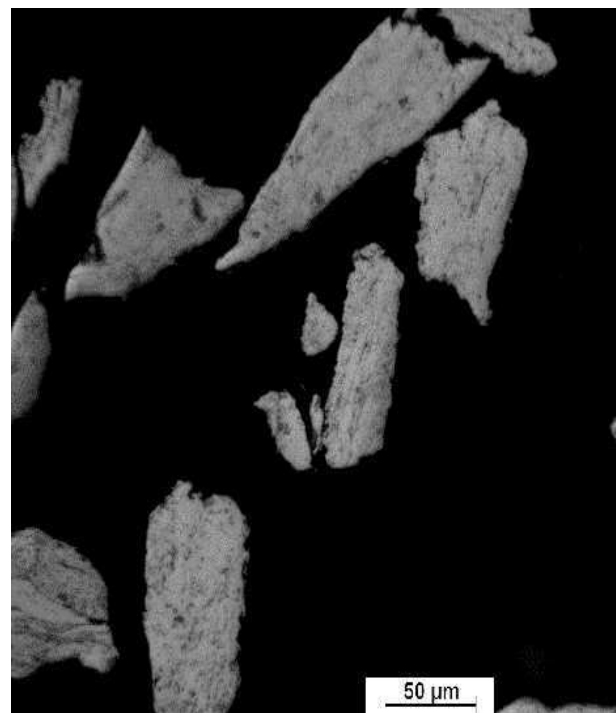


Fig. 5 Microstructure of ball-milled rapidly solidified AlFe7Mm4 ribbon (LM)

Cryo-milling of ribbons preserved the rapidly solidified microstructure, as documented in Fig. 6. As also no peak broadening was observed by XRD in Fig. 2.

Sample B1 was prepared by sintering of whole ribbons. The turbulent flow of material during pressing is visible on microstructure of B1 sample presented in Fig. 7. The sample was not sintered satisfactorily and the initial ribbons delaminated easily.

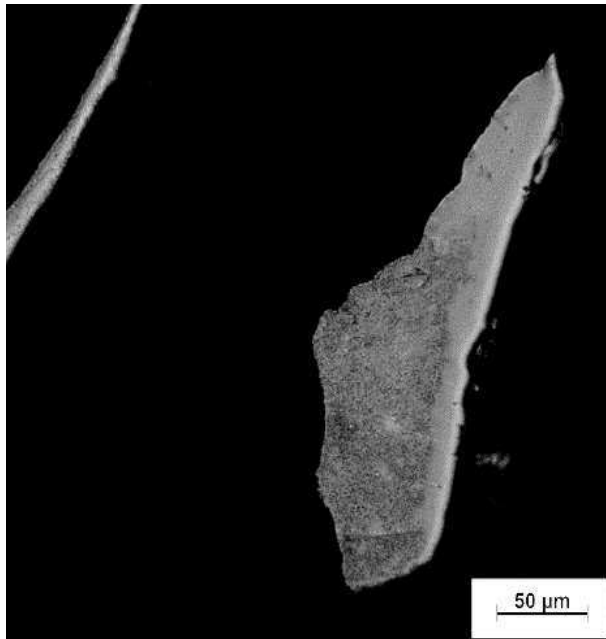


Fig. 6 Microstructure of cryo-milled rapidly solidified AlFe7Mm4 ribbon (LM)



Fig. 7 Microstructure of bulk AlFe7Mm4 alloy, B1 (LM)

Sample B2 was sintered from cryo-milled powder at 450°C. The pressure of 80 MPa at this temperature was not sufficient for sintering bulk samples without significant amount of pores, as illustrated in Fig. 8. Massive delamination took part during machining of sample.

Sample B3 was prepared from cryo-milled powder by pre-pressing of cylinder with diameter of 19 mm that was subsequently sintered at 450°C by SPS. The sintering was successful and compact bulk sample was formed. The sample contained porosity localized in one row probably resulting from pre-pressing conditions. The microstructure of sample B3 is shown in Fig. 9.

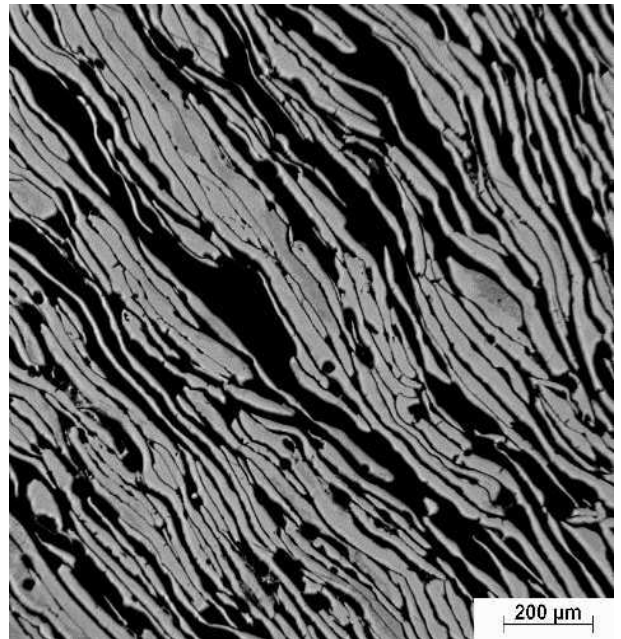


Fig. 8 Microstructure of bulk AlFe7Mm4 alloy, B2 (LM)

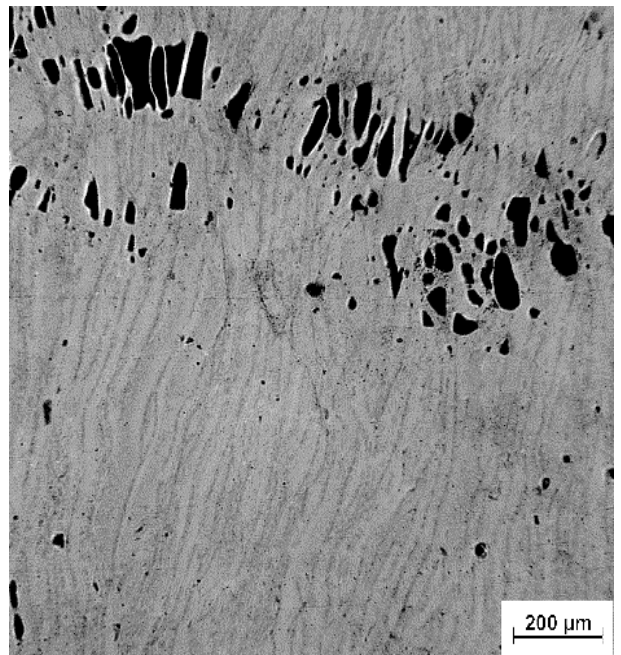


Fig. 9 Microstructure of bulk AlFe7Mm4 alloy, B3 (LM)

The influence of processing on hardness of AlFe7Mm4 alloy is plotted in Fig. 10. After melt spinning, the hardness increased due to grain refinement. Ball-milling lead to further hardness increase caused by deformation strengthening. Hardness of cryo-milled sample seems to be lower than the one of initial rapidly solidified ribbons. This might be experimental error caused by small size of powder particles. The value of hardness is combination of hardness of metal powder and embedding material. As the hardness value for porous B2 sample is similar, this explanation is highly probable. The lower value of hardness of B3 sample is likely to be caused by Al matrix grain coarsening at higher sintering temperature.

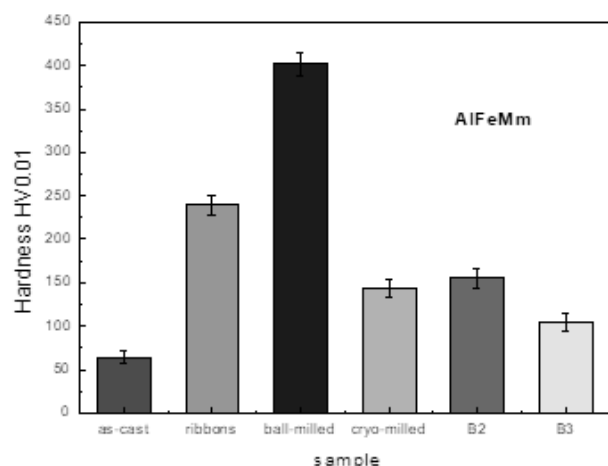


Fig. 10 Hardness HV0.01 of studied samples

4 Conclusion

Rapidly solidified ribbons of AlFe7Mm4 alloy were sintered by SPS method. It was proven that sintering of whole ribbons leads to unsuccessful sintering caused by oxide layers on the surface of ribbons. Pre-treatment of ribbons by ball-milling is not suitable because of destruction of rapidly solidified microstructure. Cryo-milling of ribbons lead to powder applicable for sintering. Temperature of 450°C was low for sintering as the prepared material was extremely porous. Sintering temperature of 500 °C seems to result in a bulk sample. Pre-pressing before sintering helps to the sintering process.

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