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Heat resistant Al-based profiles possessing high strength at elevated temperatures

M. Balog[1]*, J. Nagy[1], F. Simančík[1], K. Iždinský[1], P. Švec[2], D. Janičkovič[2]

[1]*Institute of Materials and Machine Mechanics, SAS, Račianska 75, 83102 Bratislava, Slovak Republic*
[2]*Institute of Physics, SAS, Dúbravská cesta 9, 84511 Bratislava, Slovak Republic*

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Abstract

New approach where hard-to-deform AlFe2V4 melt-spun ribbons (MSR) were compacted into sound profiles using "non-degrading" plasticizer is shown. The plasticizer, made of 1 μm Al 99.7 % powder, enabled significant reduction of extreme loads required for extrusion of plain AlFe2V4 MSR and meanwhile improved the plasticity of extruded compacts. After extrusion plasticizer forms a "matrix" part of two-phase composite structure, which is reinforced with chopped AlFe2V4 MSR flakes. No apparent phase transformation took place even after long term annealing held at 350 °C, neither within any of compact's constituents. Unique structural stability of extruded "composites" was attributed to randomly oriented icosahedral particles, with traces of an amorphous phase and fcc Al10V intermetallic phase in AlFe2V4 MSR and to efficient strengthening by homogeneously distributed nanoscale dispersoids coming from broken surface oxides of original 1 μm Al powder plasticizer particles. Room temperature ductility and toughness of mixture compacts were significantly improved in comparison to plain AlFe2V4 MSR compacts. The prepared material reproducibly attained the strength above 500 MPa at 3–4 % ductility. Outstanding mechanical properties were attained especially at elevated temperatures (ultimate tensile strength UTS ~ 280 MPa and ductility of 4–5 % at 300 °C). The suggested technological approach enables real utilization of RS ribbons in bulk structural profiles, whereas most of conventional technological equipment can be used for this purpose without need of intensive new investments.

Key words: aluminium, rapid solidification, high strength, heat resistance, powder metallurgy, melt-spinning, extrusion

1. Introduction

Potential service of high strength Al alloys at elevated temperatures (above 200 °C) cannot be fulfilled with commercial Al alloys if their high strength is based on precipitation strengthening. Herein distinctive loss of mechanical properties due to overaging effects is observed even at moderate temperatures (usually above 150 °C) [1]. Al-Fe-V alloys become very attractive in this case, because of low diffusion coefficient of iron and vanadium in aluminium and thus potentially higher structural stability of present phases at elevated temperatures [2–4]. Unfortunately, the equilibrium solubility of these elements in aluminium lattice is very low and even at high temperatures it does not exceed 0.03 at.%. The consequence is that conventional thermal treatment cannot bring any efficient strengthening in case of these alloys.

Aforementioned problem can be overcome by utilizing of rapid solidification (RS) technique, which ensures at least three interesting features [5]: significant increase of alloying elements solid solubility, profound refinement of structural grain and formation of variety non-equilibrium aperiodic phases. In case of Al-Fe-V alloys, employment of rapid solidification yields significant iron solid solubility increase (up to 4 at.%) and thus promotes the development of fine scale phases, which are stable up to relatively high temperatures simultaneously assuring high strength and stiffness within rather broad temperature range. Moreover un-

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*Corresponding author: tel.: +421 59209401; fax: +421 54772909; e-mail address: ummsbama@savba.sk

like other RS Al-based alloys, Fe and V as relatively cheap alloying elements make the alloy prospectively competitive in potential industrial applications. Typical candidate of RS Al-Fe based alloys recently intensively studied is AlFe2V4 composition [5, 6].

However, the necessary heat flow associated with rapid solidification limits at least one dimension of material, which can be cooled down at desired cooling rate. As a result, only discrete particles are attained and some kind of consolidation must be performed if bulk products are to be produced. To maintain desired RS microstructures and establish good metallurgical bonding between particles, the consolidation is basically circumscribed to compaction routes based on introduction of plastic deformation at moderate temperatures. To attain proper metallic bonds oxide layers always present at particle surfaces have to be broken what can be assured exclusively by means of shear deformation. Considerable effort has been therefore paid to consolidate rapidly solidified AlFe2V4 melt-spun ribbons (MSR) via direct extrusion [6]. However, extremely high pressing loads are required if the compaction of high strength AlFe2V4 MSR is performed at "lower" temperatures to assure maintaining of their unique RS microstructure. Such extreme pressing loads are technologically feasible only at laboratory scale. Moreover, the poor workability and lack of MSR plasticity negatively affected the material flow during extrusion and were responsible for the formation of some consolidation defects. Imperfect consolidation in hand with brittleness of MSR gave a rise to occurrence of premature brittle failure during tensile tests and low fracture toughness of MSR compacts at room temperature [6].

To improve consolidation and room temperature mechanical properties of AlFe2V4 MSR compacts new approach is suggested in this work. The idea is based on introduction of "non-degrading" plasticizer/matrix material admixed into MSR which would reduce extreme loads (or consolidation temperatures) required for direct extrusion of plain MSR and meanwhile improve the plasticity and fracture toughness of extruded compacts. Plasticizer is to structurally stable at elevated temperatures, it has to be compatible with MSR, providing sound bonding without occurence of any inferior interfacial reaction and it has to accomplish potential recycling needs.

2. Experimental

AlFe2V4 melt-spun ribbons (MSR) were produced by planar flow casting of master alloy from the temperature of about 1150 °C onto rotating copper chill providing cooling rate $\sim 10^6$ K·s^{-1}. Master alloy was prepared in vacuum induction furnace from 99.9 % pure elements. Continuous MSR with the cross sections of $\sim 0.02 \times 10$ mm were disintegrated into smaller flakes by means of "cryo-chopping", which did not deplete the plasticity of as-cast MSR [6].

Ultrafine ($d_{50} \sim 1$ μm) monocrystalline Al powder of technical purity 99.7 % was employed as plasticizer. The powder, supplied by Austrian company *MEPURA G.m.bH. Ranshofen,* was prepared by gas atomization in Ar atmosphere [7]. Grain size distribution of powder was determined by means of Helos analysis as follows: $x_{50} = 1.31$ μm, $x_{10} = 0.66$ μm, $x_{90} = 2.51$ μm. The AlFe2V4 MSR + 1 μm Al 99.7 % powder mixtures were homogenized for half an hour in a Turbula shaker prior consolidation.

Compaction of MSR-powder mixtures was realized via conventional hot direct extrusion (DE) on pneu-hydraulical press using flat face die. Extrusion ratio $R = 11:1$ was found to be an optimum to assure sufficient amount of plastic shear deformation while keeping extrusion loads technologically feasible [8, 9]. No flushing, degassing and canning operations were carried out prior to extrusion to keep the process economically acceptable. Either loose or precompacted (cold isostatic pressing at 900 MPa) MSR-powder mixtures were filled into the preheated die (50 °C below extrusion temperature) and afterwards heated up to desired compaction temperatures for 30 min before extrusion. BN spray was used as a lubricant. To prevent the particles from pouring out of the die during filling and meanwhile to decrease friction during extrusion, 2 mm thick Al coin was placed between the die and compacted material. The average ram speed during extrusion within chosen compaction temperature range was ~ 1 mm·s^{-1} ($\pm$ 0.8).

The mechanical properties at room temperature and at 300 °C (20 min to reach and stabilize desired temperature + 10 min dwell prior experiment) were measured on specimens with the gauge of ø 4–32 mm (tension) and ø 5.5–10 mm (compression) using ZWICK tensile machine at the cross ram speed of 1 mm·min^{-1}. Vickers microhardness measurements were performed on compact's cross section with applied load of 10 g. 3-point bending dynamic mechanical analysis (DMA) experiments were performed on 2980 DMA 1.7B machine at the Technical University of Vienna using at least two samples of each material with dimensions of $4 \times 1.8 \times 50$ mm. Experiments were carried out in one cycle at the heating and cooling rate of 3 °C·min^{-1} within temperature range up to 350 °C at frequency of 1 Hz and amplitude of 40 μm. Dilatometry experiments were also performed at the Technical University of Vienna using thermal-mechanical analysis equipment TMA 2940 CE, Thermal Instruments, USA, see details elsewhere [10].

Microstructures were characterized by means of light microscopy, transition electron microscopy (TEM) using the JEOL JEM 100 C electron micro-

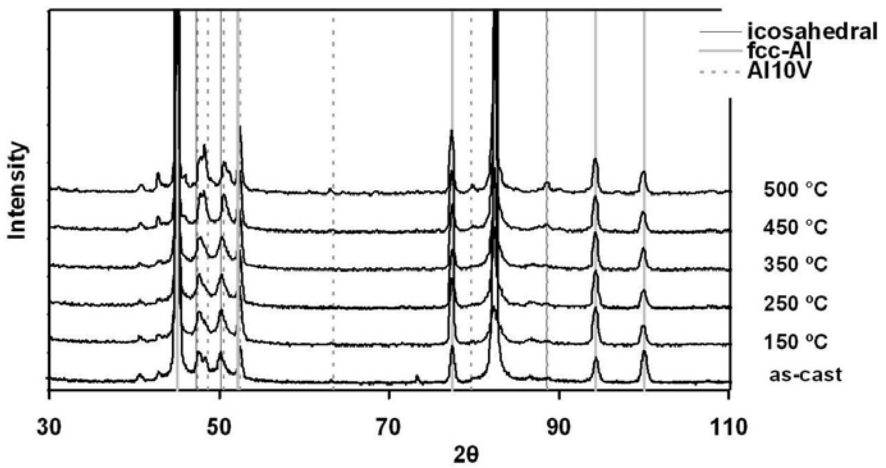

Fig. 1. XRD traces of AlFe2V4 melt-spun ribbons in as-received state and after different isothermal exposures at annealing temperatures of 150, 250, 350, 450 and 500 ℃ for 30 minutes.

scope operated at 100 kV and scanning electron microscopy (SEM) using the JEOL JSM 5310 at 20 kV. TEM foils were prepared by mechanical grinding and electrolytic etching (80 % alcohol, 10 % ethylenglycol and 10 % fluoric acid). X-ray diffraction patterns were obtained by standard XRD using Cu Kα radiation. Both isothermal and anisothermal resistivity measurements were carried out using a high precision four probe method with long time temperature stability. The ratio R/R_0 of the electrical resistivity R to that at room temperature R_0 (or at the intitial time) was used in preference to the absolute electrical resistivity, in order to avoid the effect of errors in sample size [12].

3. Results and discussion

3.1. Characterization of as-cast AlFe2V4 melt-spun ribbons

XRD diffractions patterns of as-received RS AlFe2V4 melt-spun ribbons (MSR) revealed the presence of three main structural constituents: fcc-Al phase, icosahedral and fcc $Al_{10}V$ intermetallic phases (Fig. 1). With increase of annealing temperature during isothermal exposures of as-received MSR, peaks of icosahedral phase gradually diminished on expense of peaks related to $Al_{10}V$ intermetallic phase, which appeared to be more apparent. This structural transformation became especially profound after high temperature annealing at temperatures over 450 ℃.

Devitrification of as-received AlFe2V4 MSR took place in two steps (Fig. 2). First exothermic reaction was associated with formation of quasicrystalline phase and its onset was found at ~ 320 ℃. At the temperature of ~ 480 ℃ onset of $Al_{10}V$ phase formation was confirmed.

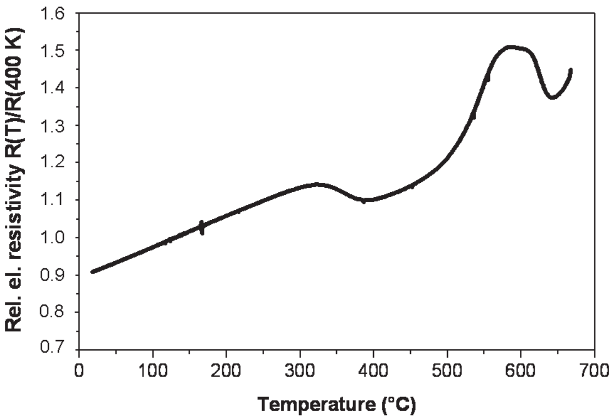

Fig. 2. Devitrification of as-received AlFe2V4 MSR (relative electrical resistivity).

TEM observations and SAED revealed that the fine grained microstructure of as-received ribbon was predominantly formed by α-Al grains and randomly oriented icosahedral particles. α-Al grains are often not perfect single crystals but contain slight misorientations. The icosahedral particles are located inside as well as in grain boundary regions of α-Al grains. Icosahedral particles are of spherical morphology with diameters mostly not exceeding the 100 nm range. Moreover, the traces of an amorphous phase as well as fcc $Al_{10}V$ intermetallic phase were confirmed to be within the MSR structure.

However, microstructures with presence of all phases were found to be quite heterogeneous. That was linked to mainly inconstant cooling rates operating in particular ribbon locations. The ribbon thickness varied in the 0–20 μm range and the cooling rates are expected to somehow reflect this scatter. Furthermore it is thought that the time for homogenization of molten alloy prior casting was insufficiently short

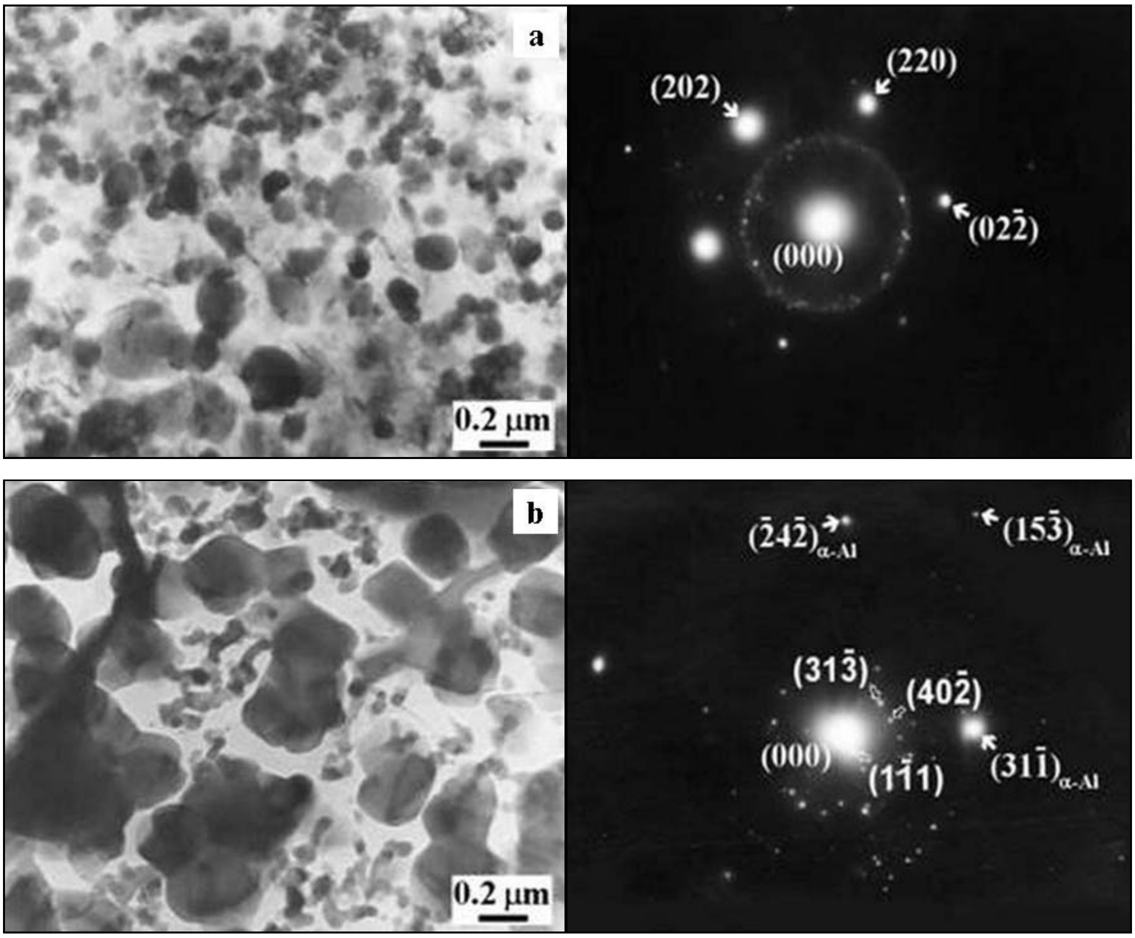

Fig. 3. TEM micrographs with corresponding selected-area diffraction patterns of AlFe2V4 melt-spun ribbon after annealing at 450 ℃ (a) and at 500 ℃ (b) held for 30 minutes.

to dissolve all coexisting phases (given by equipment hindrances).

No dramatic changes, indicating potential phase transformations, appeared in the microstructure of thermally exposed ribbons up to a temperature of ~ 450 ℃ (Fig. 3a). The diameter of icosahedral particles did not markedly grow with increasing temperature. However, the presence of an amorphous phase was not so evident in samples that had been exposed at higher temperatures. Further increase of annealing temperature to 500 ℃ gave a rise to substantial grow of intermetallic $Al_{10}V$ phase (Fig. 3b).

3.2. Consolidation of plain AlFe2V4 melt-spun ribbons

Consolidation of plain AlFe2V4 MSR was carried out using conventional hot direct extrusion (DE). The highest possible extrusion temperature (T_{ext}) was limited by onset of hard and brittle intermetallic phase $Al_{10}V$ formation. Excess of $Al_{10}V$ would significantly deplete the plasticity of MSR, it would make consolidation even more difficult and eventually it would deplete compact's ductility and toughness. Nevertheless, reproducible extrusion trails performed at $T_{\text{ext}} = 470$ ℃ were possible, although extreme value of $p_{\max} = 1560$ MPa was required (Fig. 4). Such high pressure at the level of tool strength limit is technologically feasible only at the laboratory level and is far beyond potential industrial applicability. Defragmentation of MSR flakes and their alignment into extrusion direction were responsible for sudden pressure drop of extrusion load after passing $p_{\max}$ value. No influence of the cryo-chopped MSR size fraction on $p_{\max}$ value and consolidation behaviour was observed.

Microstructural observations revealed well-deformed MSR flakes bound and arrayed into extrusion direction (Fig. 5a). Even though a very good metallurgical bonding between MSR particles was confirmed, the absence of deformation texture clearly revealed their poor plasticity. Only few traces of plastic deformation within MSR, namely twinning formation, were observed. Lack of MSR plasticity negatively affected the material flow during extrusion and was responsible for the formation of consolidation imperfections observed predominantly in the central extrusion axis

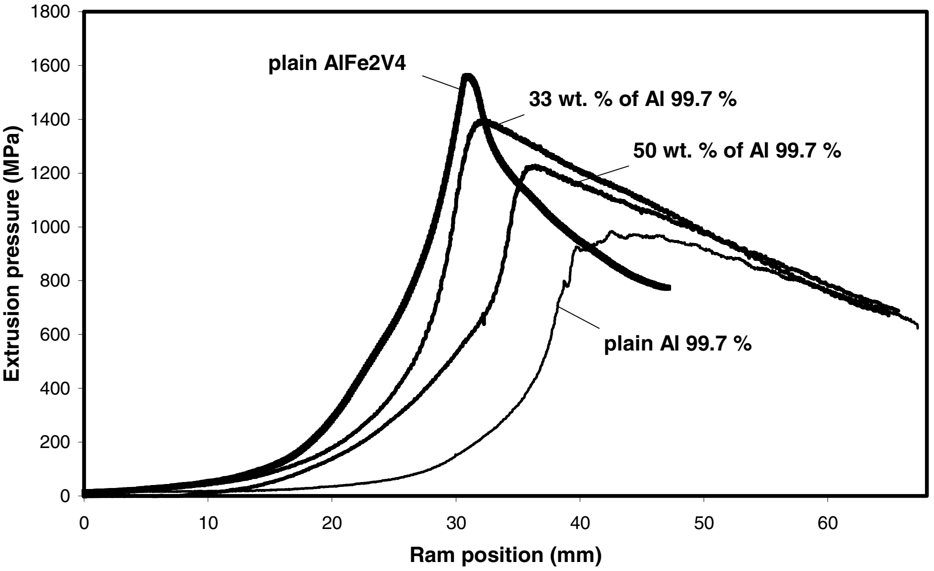

Fig. 4. Evolution of extrusion pressures recorded during consolidations of plain AlFe2V4 MSR flakes and 1 μm Al 99.7 % powder plasticizer itself. Effect of two different amount of plasticizer admixed into AlFe2V4 MSR ($T_{\rm ext}$ = 470 °C, 0–200 μm MSR fraction).

zone with less shear deformation induced (Fig. 5b). In agreement with annealing experiments performed on as-received MSR no new morphologies appeared in the compact's microstructure after consolidation.

High compression strength of AlFe2V4 MSR compacts up to 775 MPa was determined. High strength was, however, not confirmed by subsequent more valuable tensile tests. MSR compacts prematurely failed at UTS = 548 MPa during tensile tests right after reaching yielding (Fig. 6a). Herein poor compact's ductility was associated with compaction imperfections. This was obviously not the case of compression tests, less sensitive to macrostructural inhomogeneities. Outstandingly high UTS of 327 MPa accompanied with significantly enhanced ductility A = 1.8 % was reached during tensile tests carried out at 300 °C (Fig. 6b). Relatively high Young's moduli of 96.5 GPa and 77.5 GPa determined by means of DMA analyses were confirmed at RT and 300 °C, respectively.

3.3. Consolidation of AlFe2V4 melt-spun ribbons using 1 μm Al 99.7 % powder "plasticizer"

The main reason to utilize plasticizer in compaction of chopped AlFe2V4 MSR was to reduce extreme loads (or temperatures) during extrusion, improve consolidation quality without internal defects and meanwhile increase the ductility of final compacts. Ultrafine ($d_{50} \sim 1$ μm) monocrystalline Al powder of technical purity 99.7 % was employed for this purpose. Detailed study of consolidation behaviour of this powder is given elsewhere [11].

From Fig. 4 it can be seen how plasticizer admixed

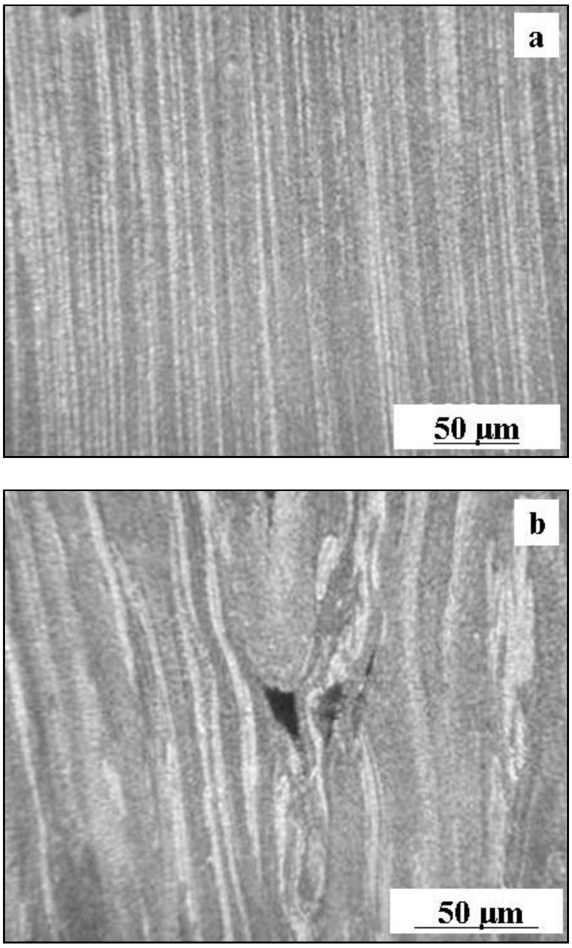

Fig. 5. Longitudinal cross-sections of compact extruded of AlFe2V4 melt-spun ribbons at $T_{\rm ext}$ = 470 °C. Outer (a) and central (b) regions of extrudate.

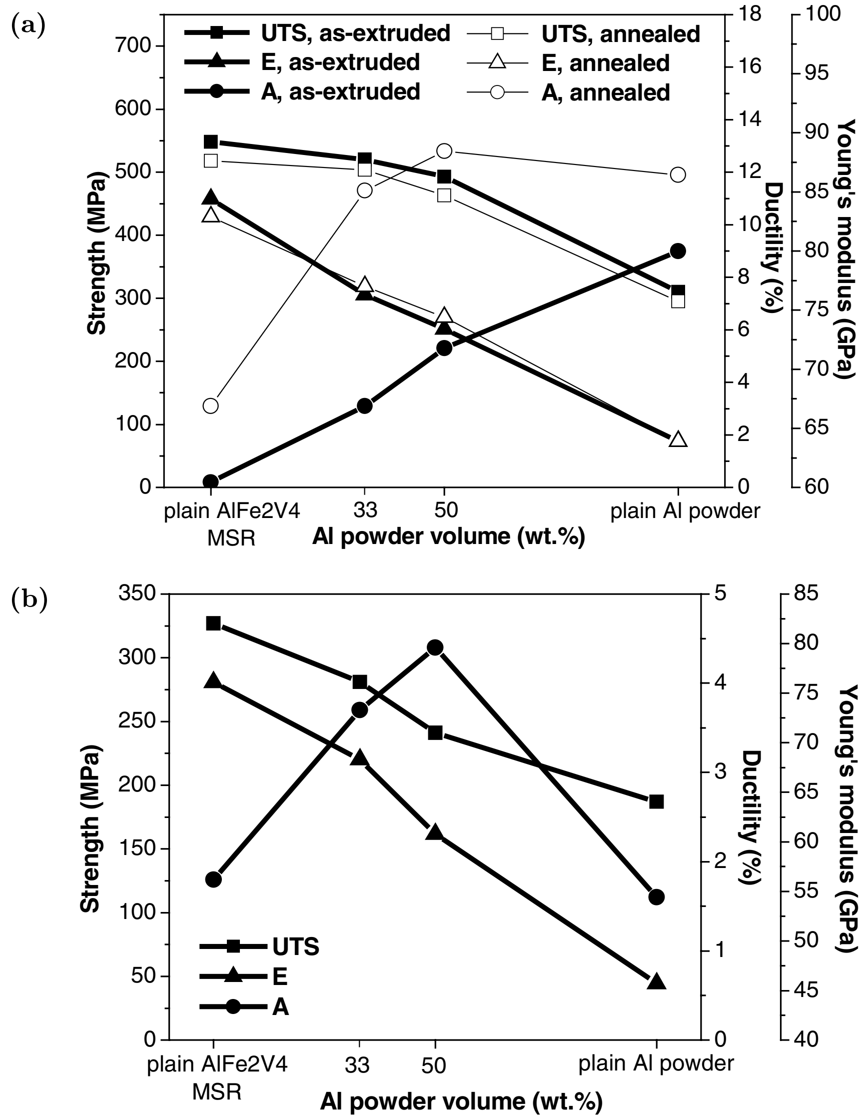

Fig. 6. Evolution of ultimate tensile strength UTS, ductility A and Young's modulus E of extruded AlFe2V4 MSR mixture compacts in hand with increasing 1 μm Al 99.7 % powder content. Obtained from tensile tests performed at room temperature (a) and at testing temperature of 300 °C (b). Effect of annealing held at 350 °C for 20 hours on room temperature mechanical properties of as-extruded AlFe2V4 MSR mixture compacts is shown in (a) ($T_{\text{ext}} = 470$ °C, 0–200 μm MSR fraction, plain 1 μm Al 99.7 % powder compacts extruded at $T_{\text{ext}} = 350$ °C).

into AlFe2V4 MSR lowered pressure peak $p_{\max}$ during extrusions at the same extrusion temperature $T_{\text{ext}} = 470$ °C. Ductile Al powder particles gave a rise to easier material flow during extrusion of mixtures. Unlike consolidation of plain MSR no distinctive pressure drop after reaching breakthrough pressure was observed. Similarly to consolidation of plain 1 μm Al 99.7 % powder, gradual pressure decrease was attributed only to descent of friction between deformed material and container walls. Drop of the $p_{\max}$ value of extruded mixtures approximately followed simple rule of mixture. 11 % drop of $p_{\max}$ was achieved by addition of 33 wt.% of Al 99.7 % powder during extrusion of mixtures held at $T_{\text{ext}} = 470$ °C. Further increase of plasticizers amount to 50 wt.% led to gradual decrease of $p_{\max}$ to the value of 1225 MPa, what represented 27 % pressure drop. While the minimum temperature of 470 °C was needed for breakthrough of plain AlFe2V4 MSR, the powder Al 99.7 % plasticizer required only $T_{\text{ext}} = 350$ °C. Leaving the $p_{\max}$ of constant value one is able to extrude MSR/plasticizers mixtures at lower extrusion temperatures and thus maintain the unique rapidly solidified structure of AlFe2V4 MSR microstructures without significant changes. For case of 33 and 50 wt.% of powder plasticizers the lowest feasible T_{ext} of 450 °C and 420 °C were obtained, respectively.

Figure 7 demonstrates homogenous transversal and longitudinal redistribution of cryogenically chopped MSR flakes (areas bright in contrast) arrayed along extrusion direction within Al 99.7 % powder matrix

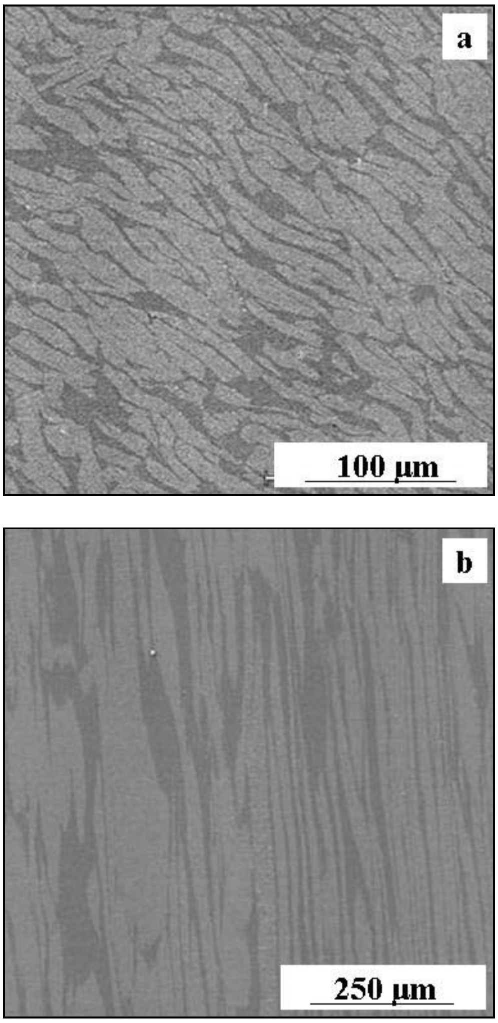

Fig. 7. Transversal (a) and longitudinal (b) cross-section micrographs of AlFe2V4 MSR + 1 μm Al mixture compacts (63–200 μm MSR fraction, 33 wt.% of 1 μm Al powder, $T_{\text{ext}} = 470\,^{\circ}\text{C}$).

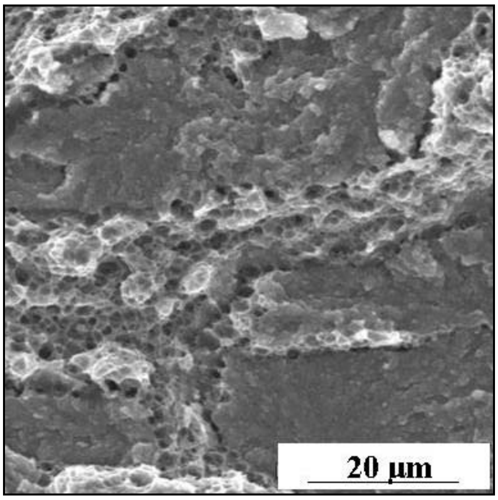

Fig. 8. Fracture surface after room temperature tensile test of AlFe2V4 MSR + 1 μm Al mixture compact (63–200 μm MSR fraction, 33 wt.% of 1 μm Al powder, $T_{\text{ext}} = 470\,^{\circ}\text{C}$).

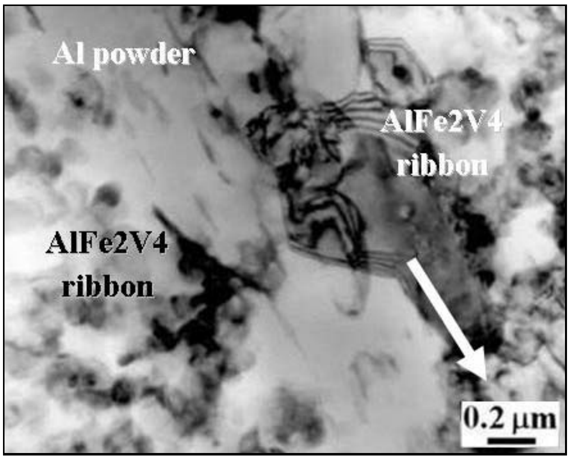

Fig. 9. TEM micrograph of AlFe2V4 MSR + 1 μm Al 99.7 % powder mixture compact. Bright field image of neighboring constituents (63–200 μm, 33 wt.% of Al powder, $T_{\text{ext}} = 470\,^{\circ}\text{C}$, arrow indicates the extrusion direction).

(dark in contrast). No apparent cracks and delamination appeared at interfaces due to constituents CTE mismatch as compacts cooled down after extrusion. Interparticle fracture through MSR flakes with no pulling-out of MSR particles and no separation at the ribbon-powder interface was detected after tensile loading (Fig. 8). That confirmed perfect metallurgical bonding on MSR/powder interfaces (Fig. 9) and hence proper transfer of stresses from matrix to strengthening MSR particles. Dimpled type fracture was observed for ductile powder areas whereas MSR fractured in obvious brittle manner. The introduction of plasticizer did not have any negative effect on structural stability of AlFe2V4 MSR. No apparent phase transformation took place after extrusion and even after long term annealing held at 350 °C for 20 hours, neither within any of compact's constituents. That was in good agreement with annealing experiments performed on as-cast AlFe2V4 MSR, plain MSR and Al powder compacts [11].

In case of 1 μm Al 99.7 % powder matrix, shearing of initially spherical powder particles during extrusion led to enlargement of particle's surface resulting in breakage of surface oxide envelopes. Torn oxide fragments were homogenously dispersed within ultrafine grained Al substrate. They were predominantly found at elongated initial particle's boundaries with transversal grain size of ~ 0.2 μm. Owing to relatively large surface area of starting 1 μm Al powder and to proper plastic deformation (breakage and redistribution of surface oxides), nano-scale oxide's dispersions significantly strengthened the microstructure of powder matrix (Fig. 9). Furthermore they acted as grain-pinning barriers preventing the microstructure against grain growth at elevated temperatures. Thus, those relatively easily introduced dispersions made use of ultra-fine grained powder microstructures during the service at high temperatures (up to 450 ℃) with no distinctive loss of mechanical properties.

In contrast with extrusion of plain MSR no traces of plastic deformation within ribbon areas were observed. That proved the role of plasticizer wherein MSR flakes were only dragged and arrayed into extrusion direction, staying only moderately deformed.

Room temperature ductility and fracture toughness of mixture compacts were significantly improved in comparison with plain AlFe2V4 MSR compacts. Ultimate tensile strength UTS = 534 MPa of mixture compacts with 33 wt.% of Al powder approached strength (548 MPa) of prematurely fractured extruded plain ribbons (Fig. 6a). However, in this case the strength increase was accompanied with improved 3.1 % ductility (A). Further addition of Al powder to 50 wt.% resulted in UTS = 493 MPa, although compensated with further improvement in ductility A = 5.3 %. Clear deviation from linear course of UTS as Al powder amount increase took place at around 50 wt.%. Up to this point UTS of composites seemed to follow simple rule of mixture. Yield strength and ductility seem to follow almost linear tendencies as a function of plasticizer content in whole examined range. If this tendency is extrapolated also for UTS it is expected that UTS of plain AlFe2V4 MSR compacts can reach ~ 680 MPa. Unlike large discrepancy between tensile and compression strength of extruded plain ribbons due to insufficient consolidation, good correlation between tensile and compression tests pointed out to successful consolidation of mixture compacts. Compressive strengths of 553 MPa and 514 MPa were confirmed at 2 % of compressive strain for 33 and 50 wt.% of Al powder, respectively.

Outstanding high temperature strength of plain MSR compacts was retained also in case of mixture compacts, where addition of plasticizer led to only marginal strength decline though at bettered ductility. Promising UTS = 281 MPa and 241 MPa accompanied with good A = 3.7 % and 4.4 % were obtained for mixture compacts tested at 300 ℃ of 33 wt.% and 50 wt.% contents of 1 μm Al powder, respectively (Fig. 6b).

As determined by means of DMA evolution of Young's storage modulus E with testing temperature of mixture compacts containing 33 and 50 wt.% of AlFe2V4 MSR showed linear tendency. Relatively high E of 82.6 GPa and 63.4 GPa were found out for 50 wt.% powder content at RT and 300 ℃, respectively. With respect to determined E, mixture compacts tended to follow simple rule of mixture.

Annealing held at 350 ℃ for 20 hours did not accommodate major changes in mixture structures. Even though no major phase transformations and microstructural changes were observed, annealing led to softening of MSR and slight fall in room temperature UTS of mixtures as seen in Fig. 6a. Significant increase in ductility of as-annealed mixture compacts took place. This is believed to be attributed both to softening of as-annealed MSR as well as to improvement of interfacial bonding between matrix and MSR due to diffusion processes accommodated during long term annealing.

4. Conclusions

It has been shown that rapidly solidified AlFe2V4 MSR could be successfully compacted into sound profiles via direct extrusion with improved consolidation behaviour and compact's mechanical properties, if fine grained 1 μm Al 99.7 % powder was used as plasticizer.

– plasticizer reduced extreme loads (or T_{ext}) required for DE of plain MSR and meanwhile enhanced the plasticity and fracture toughness of extruded mixture compacts

– after compaction, plasticizer formed a "matrix" part of two-phase composite structure, reinforced with MSR flakes

– intense work induced during extrusion resulted in ultra-fine grained Al powder matrix strengthened and grain pinned with superiorly defragmented and distributed nanoscale oxides dispersoids originated from torn surface oxide envelopes

– no delaminating or pulling out took place at matrix – MSR boundaries and perfect metallurgical interfacial bonding was confirmed

– no apparent phase transformation took place after extrusion and after long term annealing held at 350 ℃ for 20 hours, neither within any of compact's constituents

– room temperature UTS = 534 MPa of mixture compacts was found to be at the level of plain MSR compacts however accompanied with improved A = 3.1 % (for 33 wt.% of Al powder)

– high UTS = 281 MPa along with good A = 3.7 % were confirmed by tests held at 300 ℃ for mixture compacts (for 33 wt.% of Al powder)

The suggested technological approach enables real utilization of RS ribbons in bulk structural profiles, whereas most of conventional technological equipment can be used for this purpose without need of intensive new investments.

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