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PHASE EVOLUTION IN RAPIDLY SOLIDIFIED Al-Fe-V-Si ALLOYS AT CHANGES OF MAIN COMPONENTS RATIO

Al alloys for high-temperature applications are of substantial interest. Adding of transitional elements leads to precipitation of stabile multi-component inter-metallic compounds with low coursing rate at elevated temperatures [1; 2]. Such alloys can be prepared by rapid solidification from the melt which leads to increased solubility of alloying components and diminishing of grain structure and the size of the inter-metallic compounds [3; 4]. The temperature stability of this microstructure is a result of both low solubility of the inter-metallic phase in Al – matrix and retarded diffusion of the main components. Alloys from Al-Fe-V-Si are of particular interest and specially with the composition: Fe – 8,5 wt.%, V – 1,3 wt.% Si –1,7 wt.% [1]. These alloys are lightweight, with elevated tensile strength (σ_B), good corrosion resistance and easy machining. The structure of these alloys can be modified by changing the concentrations of main components or adding new ones and it is the purpose of this work to study their structure formation.

Experimental

Microcrystalline ribbons (of 50-70μm thickness and 10 mm width) were obtained by Planar Flow Casting (PFC). The composition, given in Table1, was found with the aid of scanning electron microscopy (JEOL-JSM 35 CF) with microprobe TRACOR NORTHERN TN-2000 and JEOL etalons.

Table 1

Composition of the studied microcrystalline ribbons (wt. %)

Ribbon	Fe	V	Si	Mg	Al
440	8,43	1	1,65	0,10	88,82
904	8,12	1,84	9,32	0,20	80,52
909	8,64	1,63	7,42	0,98	81,33

The microstructures were studied by optical microscopy (Richert MeF-2).

Phase transitions during heating were monitored by differential calorimeter DSC 204 Phoenix (NETZSCH).

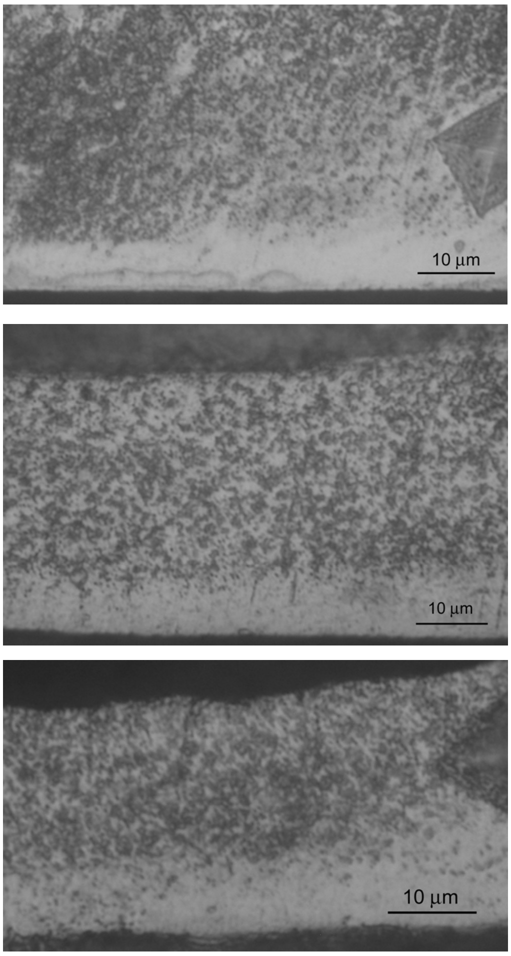
Phase identification was performed by X – ray diffractometer DRON 3 using Cu-K_α line.

Micro-hardness was measured by NEOFOT microscope and Haneman apparatus with 0.025 kg for 20 sec load.

Results

Two regions: nano – on the side of the quenching disk and micro- on the opposite side can be observed in the cross-section image of the microcrystalline ribbons (fig.1). The nano-crystalline region varies between 5 to 20 μm depending on the cooling velocity, dimension of jet nozzle and composition. Structural changes were observed mainly in the nano-region when the ribbons were heated at temperature necessary for their extrusion into bulk ingots. The

microstructure in this region becomes visible with optical microscope after annealing at 400 ^{o}C. The same picture was observed in all studied ribbon samples from Table 1.



1. As-cast ribbon 440

*2. Ribbon 440 annealed at 100^{o}C for 60 m*in

3- Ribbon 440 annealed at 400^{o}C for 60 min

Fig. 1 Microstructure of ribbon 440, 1- as-cast, 2 and 3 annealed for 60 min at 100^{o} and 400^{o}C.

3 phases were identified in the microcrystalline ribbons by X –ray analysis – Al solid solution, Si particles, the inter-metallic phase – $Al_{13}(Fe,V)_3Si$. The quantity and size of the phases is shown in Table 2. As is seen the size of both silicon and inter-metallic phase varies between 15 and 20 nm.

Table 2 Phase composition in the nano-region of as-cast ribbons 904 and 909

Ribbon	Al matrix	Si particles	$Al_{13}(Fe,V)_3Si$ phase
904	70.1 wt. %- 37.2 nm Lattice par. 4.0481 Å	4.1 wt. %- 18.5 nm	25.9 wt. %- 20.1 nm
909	-	4.7 wt. %- 14.9 nm	27.2 wt. %- 15.8 nm

It was found that Al-Fe-Si-V alloys with Si content less then 3 wt. % consist of Al – matrix and precipitated inter-metallic phase [1]. Ribbon 440 from Table 1 is of similar composition and hence has the same phase composition of 2 phases only.

In the nano- region of the ribbons the lattice parameter of Al matrix has lower value (Table 2) than the equilibrium value of pure Al (4.0493 Å) and that implies supersaturated solid solution prone to decomposition at annealing. Similar results [5] showed variation of Al - lattice parameter in microcrystalline Al-Si –Fe ribbons between 4.04314Å and 4.04575Å depending on alloy composition.

A DSC curve of ribbon 440 (low Si content ribbon) heated with 20 K/min is shown in fig. 2.

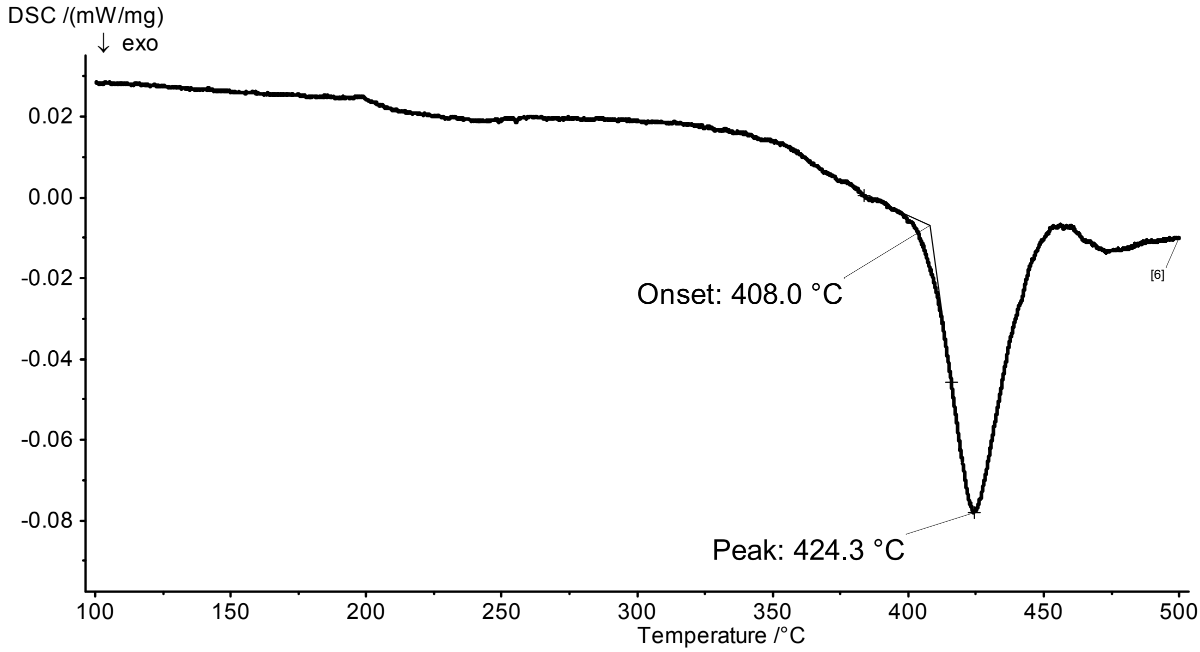


Fig. 2 DSC-curve of ribbon 440 with low Si content ribbon heated with 20 K/min

The single exothermic peak can be attributed to the inter-metallic phase ($Al_{13}(Fe,V)_3Si$) precipitation. This two phase material could be considered as a composite of Al matrix and reinforcing $Al_{13}(Fe,V)_3Si$ phase. After heating the super saturation of Al matrix vanishes and the lattice parameter becomes closer to the equilibrium value.

The micro-hardness values changes of ribbon 440 with heating are shown in fig. 3. The measurings are taken after isothermal heating for 60 min. The values go down to a minimum and then increase owning to the inter-metallic compound precipitation.

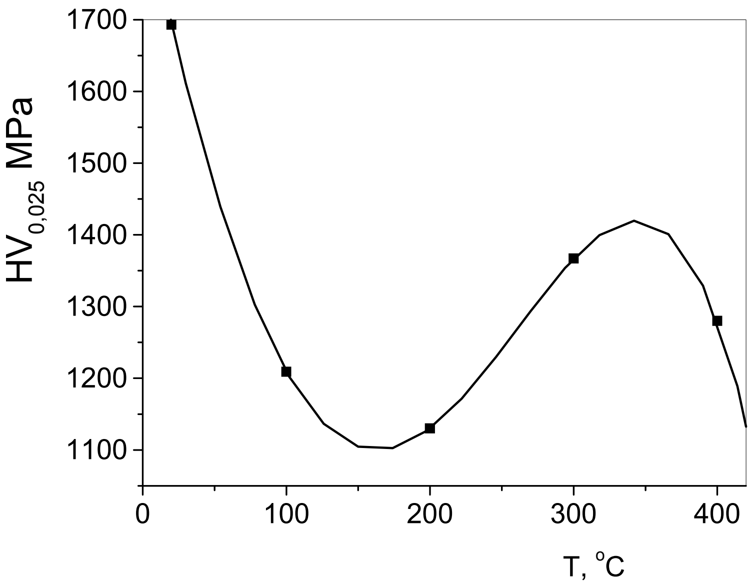


Fig 3 Micro-hardness of ribbon 440 taken after isothermal heating for 60 min.

The onset temperatures of DSC curves lie between 389.3–421.9 ^{o}C (for heating rate 5, 10, 20 and 50 K/min) which coincides well with the temperature interval of the maximum

from fig.3, hence the increase of micro-hardness is connected with inter-metallic precipitation.

DSC – curves for different heating rates (for 5, 10, 20 and 50 K/min.) are shown in fig. 4. Higher velocity leads to increase of the precipitation temperature. As this increases the thermal stability of the ribbons it can be taken into account in the annealing during hot-extrusion.

Kissinger plot [6, 7] was used to determine the activation energy of the precipitating process. The result for ribbon 440 was found to be 218.0 kJ/ mol (Table 3).

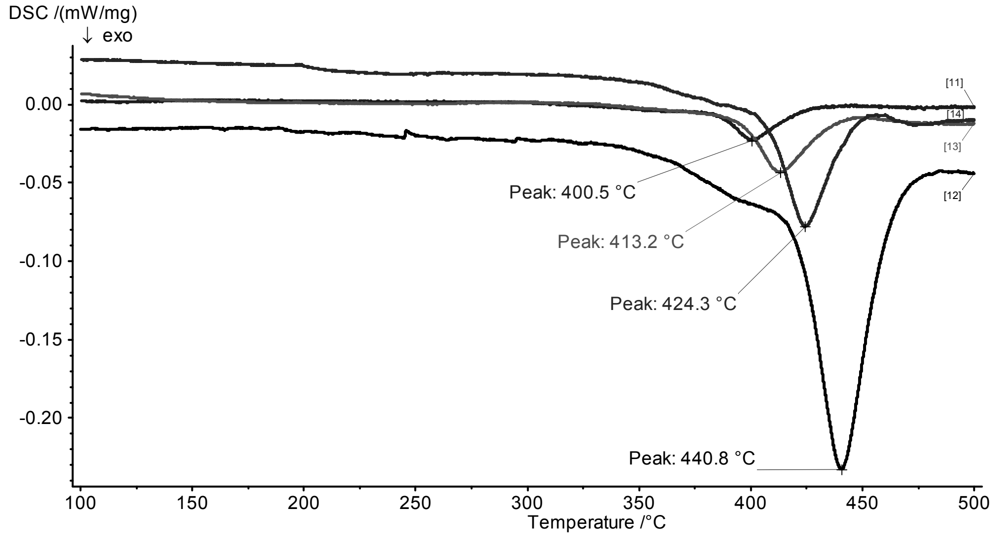


Fig. 4: DSC- curves for ribbon 440 with low Si content for heating rates 5 K/min, 10 K/min, 20 K/min and 50 K/min.

Fig. 5 shows the DSC – curve for ribbon 904 with higher Si content.

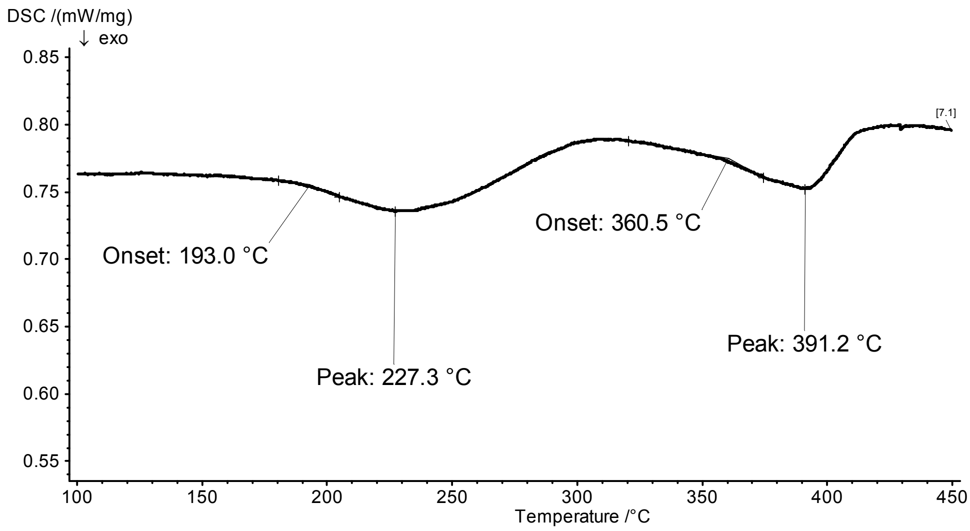


Fig. 5 DSC- curve of ribbon 904 with high Si content heated with 20 K/min

Higher Si content leads to occurrence of a second peak at lower temperature. The two peaks appear one after the other at 236.2 and 391.2 ^{o}C. The second peak which appears at about similar temperature (~400 ^{o}C) can be ascribed to inter metallic phase $Al_{13}(Fe,V)_3Si$

precipitation. The activation energies, derived from Kissinger plot of the two peaks (Table 3) are 106.6 and 260.6 kJ/mol. The activation energy for the second peak is very close to the one of ribbon 440 with 218.0 kJ/mol, hence the second peak definitely is a result of inter-metallic compound precipitation. The activation energy of the first peak is close to the one for Si precipitation in Al-Si rapidly solidified ribbons [5]. It can be assumed that this peek corresponds to precipitation of secondary Si phase. Similar results were reported elsewhere [8]. X-ray analysis (Table 2) shows coexistence of tree phases in the microstructure of this ribbon– Al matrix, Si particles and an inter-metallic compound.

The nano- size, precipitated Si and inter-metallic particles in ribbons could not be distinguished by optical or electron microscope. Therefore the phase identification was performed only on hot-extruded samples of the ribbons as their structure become coarser and hence observable. In fig. 6 a microstructure of hot-extruded sample of ribbon 904 obtained by an optical microscope is shown. A tree phase structure can be seen. In fig. 7 the same sample was studied by EDAX- microprobe and the Si and inter-metallic phases can clearly be distinguished [9].

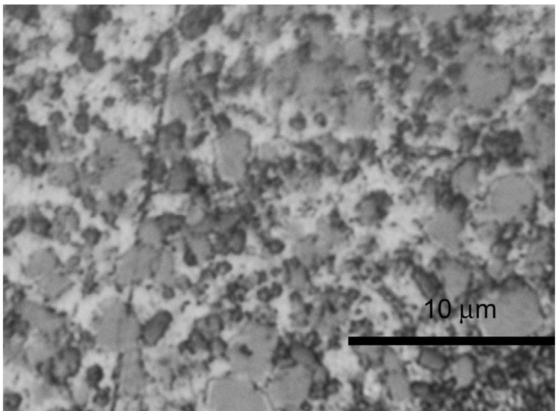


Fig. 6 Microstructure of the hot- extruded ribbon 904 at 450-480 ^oC, obtained by an optical microscope

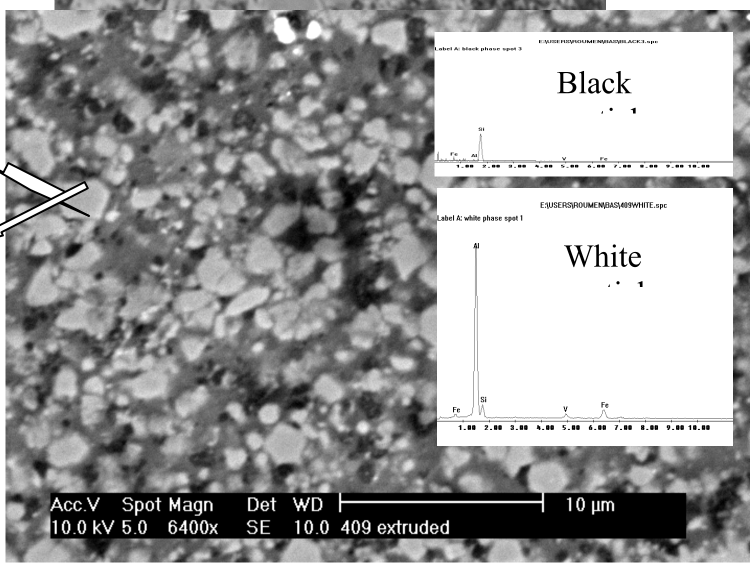


Fig. 7 Microstructure of the hot- extruded ribbon 904 at 450-480 ^oC, obtained by the Scanning Electron Microscope [9]

Ribbons of high Si content (as well as additionally hot-extruded ones) can be regarded as two reinforced particles “in situ” composites. The microhardness measurements show two hardness peaks with increasing temperature (fig. 8) [10]. These peaks can be linked to the two-phase precipitation in the ribbons 904 as shown in fig. 5.

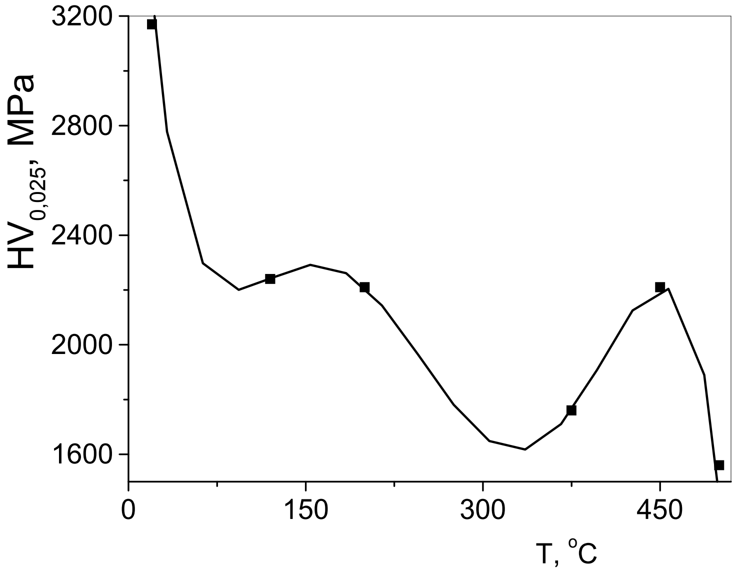


Fig. 8 Micro-hardness of ribbon 904 [10] taken after isothermal heating for 60 min.

Addition of Mg is reported to improve mechanical properties of the ribbons Al-Fe-V-Si [11]. DSC – curve of ribbon 909 having high Si content and Mg additive is shown in fig. 9.

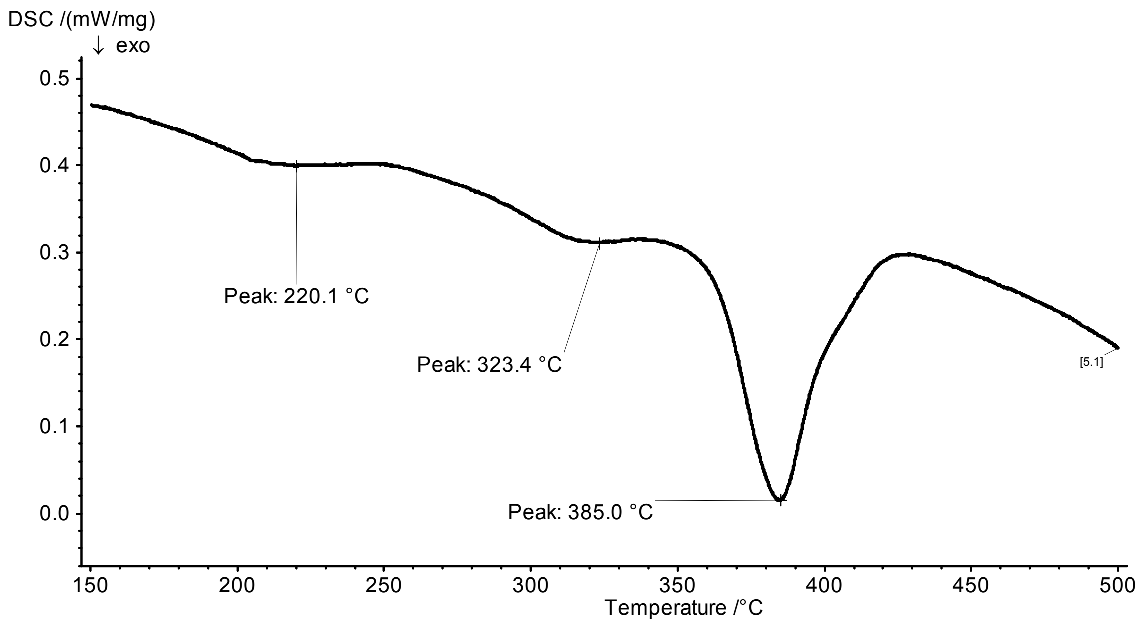


Fig. 9 DSC-curve of ribbon 909 with high Si content and Mg addition heated with 20 K/min

Three exothermal peaks can be observed where the first and the last coincide with the peaks from fig. 5. They have similar temperatures and activation energies obtained from Kissinger plot are close as well (Table 3). Hence, they result from Si and inter-metallic phase precipitations. The peak in between at about 320 °C can be attributed to inter-metallic Mg containing phase.

Table 3

Activation energies of precipitation of Si and inter-metallic $Al_{13}(Fe,V)_3Si$ phases.

Alloy	E, I peak, kJ/mol	E, II peak, kJ/mol
440	-	218
904	105,6	260,6
909	124	274

The presence of three reinforcing particles in the ribbon 909 leads to substantial increase of the micro-hardness, comparing to ribbons 440 and 904, reinforced with one and two particles respectively (Fig. 10).

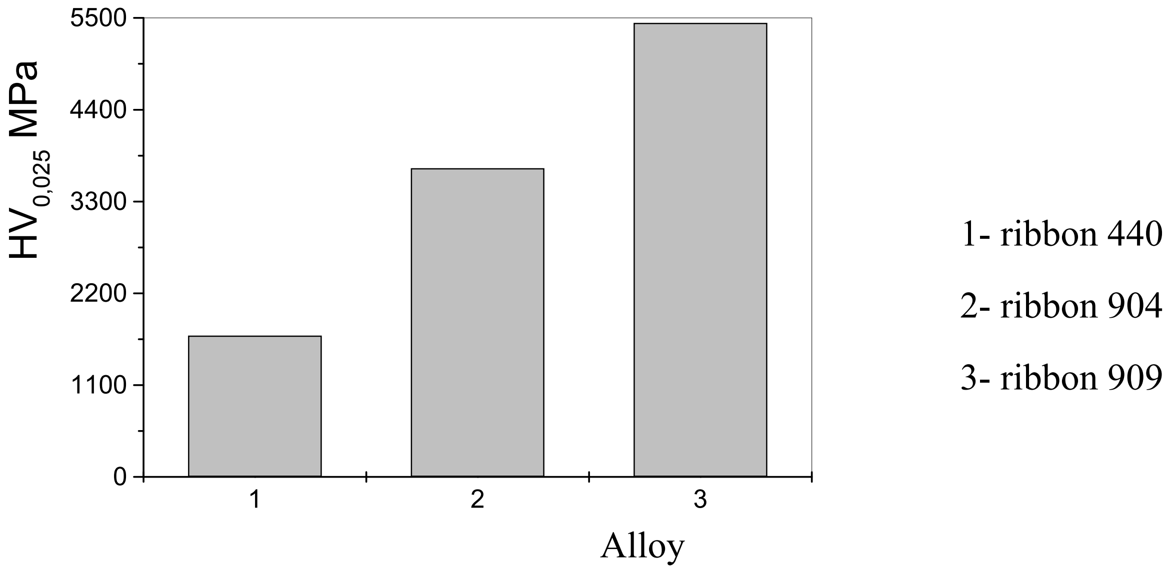


Fig. 10 Micro-hardness of ribbons 1- 440, 2 -904 and 3- 909

Tensile strength and micro-hardness were measured on samples of hot-extruded ribbons (Table 4). Instead of ribbon 909, rapidly solidified, atomized powder with the same composition was used. As is seen σ_B goes up in accordance to micro-hardness increase of the three studied ribbons as shown in fig. 10.

Table 4

Tensile strength of hot-extruded samples

Alloy	σ_B, MPa	δ, %	$HV_{0.025}$, MPa
Ribbon 440 with low Si content	300	6	1060
Ribbon 904 with high Si content	390	2	1900
Powder with high Si content and 1,1 wt. % Mg additives	430	1	1520

Summary

Microcrystalline ribbons Al-Fe-V-Si with Si content below 3 wt.% consist of two phases – Al matrix and inter- metallic $Al_{13}(Fe,V)_3Si$ phase. The increase of Si content (keeping similar Fe and V concentrations) leads to secondary precipitation of Si particles. Adding about 1wt Mg forms additional Mg containing phase.

These studied micro-crystalline alloys can be regarded as "in situ" composites reinforced with one, two and three types particles respectively and hence increases their micro-hardness. This reflects in increased tensile strength of hot-extruded ribbons in direction low Si – higher Si – higher Si + Mg alloys.

Rapidly solidified ribbons exhibit a mixture of nano- and micro -structures. The main structural changes during heating take place in the nano- region. From the observed structural changes and micro-hardness in the studied ribbons one can work out the structure and mechanical properties of the hot-extruded ingots prepared from these ribbons.

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