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MICROSTRUCTURES OF THE Al-Fe-Cu-X ALLOYS PREPARED AT VARIOUS SOLIDIFICATION RATES

MIKROSTRUKTURA ZLITIN Al-Fe-Cu-X PO RAZLIČNIH HITROSTIH STRJEVANJA

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Aluminium alloys are usually prepared with conventional casting, but rapid-solidification methods lead to the alloys with better mechanical properties or thermal stability. When an improved thermal stability is required, aluminium is alloyed with one or more of the elements from the group of transition metals (TM), for example, Ni, Fe, Cr or Ti. These elements are characterized by a low diffusivity and solubility in aluminium even at elevated temperatures, while Cu in an alloy forms a $CuAl_2$ phase that enables precipitation hardening. In this work, the microstructures of the Al-7Fe-4Cu, Al-4Fe-4Cu-3Ni and Al-7Fe-4Cu-3Cr (mass fraction, *w*/%) alloys prepared with various solidification processes were investigated. The aim of this work was to determine the changes in the microstructure caused by the increasing solidification rate and to determine the influence of the copper present in each alloy. All the samples were prepared with single-roll melt spinning, water quenching of the melt and conventional casting. The microstructures of the alloys were studied with light and scanning electron microscopy (SEM). The phase composition was determined with X- ray diffraction (XRD). Vickers hardness (HV 5) and microhardness (HV 0.005) were measured to compare the mechanical properties of the alloys. The microstructure and the hardness of the alloys strongly depended on the solidification rate. The fine microstructure and high microhardness values obtained with melt spinning are promising for the use of these alloys in special applications at elevated temperatures.

Keywords: aluminium alloy, rapid solidification, melt spinning, transition metals, microstructure

Zlitine aluminija se najpogosteje izdelujejo z navadnim ulivanjem, vendar pa metode hitrega strjevanja povzročijo nastanek zlitin z boljšimi mehanskimi lastnostmi ali toplotno stabilnostjo. Kadar se zahteva toplotna stabilnost, se aluminij legira z enim ali dvema elementoma iz skupine prehodnih kovin (TM), na primer: Ni, Fe, Cr ali Ti. Značilno za te elemente je majhna difuzivnost in topnost v aluminiju celo pri povišanih temperaturah, medtem ko Cu v zlitini tvori fazo $CuAl_2$, ki omogoča izločevalno utrjanje. V tem delu so preiskovane mikrostrukture zlitin Al-7Fe-4Cu, Al-4Fe-4Cu-3Ni in Al-7Fe-4Cu-3Cr (masni deleži, *w*/%), pripravljenih z različnimi postopki strjevanja. Namen tega dela je bil opredeliti razlike v mikrostrukturi, ki jih povzroči povečanje hitrosti strjevanja, in opredeliti vpliv bakra v vsaki od navedenih zlitin. Vsi vzorci so bili pripravljeni z ulivanjem tankega traku na bakren valj, z ohlajanjem v vodi in z navadnim ulivanjem. Mikrostruktura zlitin je bila pregledana s svetlobnim mikroskopom in z vrstičnim elektronskim mikroskopom (SEM). Sestava faz je bila določena z rentgensko difrakcijo (XRD). Trdota HV 5 in mikrotrdota HV 0,005 sta bili izmerjeni za primerjavo z mehanskimi lastnostmi zlitin. Mikrostruktura in trdota zlitin sta močno odvisni od hitrosti strjevanja. Drobnozrnata mikrostruktura in velika mikrotrdota, dobljeni z ulivanjem na valj iz bakra, sta obetajoči za uporabo teh zlitin v posebnih primerih pri povišanih temperaturah.

Ključne besede: zlitina aluminija, hitro strjevanje, ulivanje na bakreni valj, prehodne kovine, mikrostruktura

1 INTRODUCTION

Aluminium alloys processed with the conventional technologies, such as casting and forming, are widely used in many technical branches such as the aerospace and automotive industries. The main advantages of aluminium alloys are price, good strength-to-weight ratio, good castability, formability or the ability of precipitation hardening. However, the mechanical properties of traditional alloys made of Zn, Mg or Cu strongly degrade at elevated temperatures, which means that their application is then limited to 150–200 °C. One way of improving the thermal stability of aluminium alloys is to use the elements from the transition metals group (TM). Transition metals, such as Ni, Fe, Cr or Mo, are characterized by a low diffusivity and solubility in aluminium even at elevated temperatures and they are able to stabilize the materials properties up to relatively high temperatures (about 400 °C). Cu is used as an alloying element to increase both the strength and the hardness due to the $CuAl_2$ phase that allows precipitation hardening of the material.[1] Conventional casting processes produce the alloys containing coarse particles of hard and brittle Al-TM intermetallic phases, degrading the mechanical properties.[2] Therefore, it is desirable to keep these alloying elements dissolved in the matrix or in the finely dispersed intermetallic particles. A fine microstructure can be obtained by increasing the solidification rate, e.g., by atomisation or melt spinning.[3,4]

The alloying elements mentioned above are often the contaminants of Al scrap. In recent years, the consumption of aluminium alloys in engineering has been rising, causing the problems of recycling and waste disposal. Al scrap is never only pure aluminium, but it is mixed with steel, cast iron, copper alloys, etc. Parts of ferromagnetic iron-based alloys can be separated using magnetic separation. The other way is to dilute the melt with pure aluminium, but this technique increases the cost of recycled aluminium alloys. In general, the transition elements included, e.g., in the austenitic-stainless-steel admixtures in Al scrap are very difficult and costly to remove. We would like to develop a new way of preparing the alloys with interesting mechanical properties and a thermal stability using this contaminated Al scrap. The manufactured alloys would have better properties such as hardness, thermal stability and ductility associated with the low density. Due to the mentioned properties these alloys could be able to replace titanium alloys in some applications, while their price and density would be lower.

This work describes the microstructure and properties of the Al-Fe-Cu-X alloys prepared at various cooling rates. These alloys simulate the real alloys originating from melting the contaminated Al scrap. The alloys with the mentioned chemical composition have not been studied yet; there are only a few studies dealing with the microstructures of the rapidly solidified ternary alloys or systems of the quasicrystal chemical compositions.[1,5–8]

2 MELT-SPINNING PRINCIPLES

Atomization of a melt with an inert gas or water produces the powders that solidify with the rate ranging from 10^2–10^4 K s^{-1}. Melt spinning allows even higher cooling rates (10^4–10^6 K s^{-1}). In this process, a molten alloy is ejected on a high-speed rotating metallic wheel. The alloy solidifies rapidly in contact with the wheel. This method produces thin ribbons, whose thickness varies in the order of ten micrometres. Due to rapid-solidification processes transition metals can be added to aluminium even above their equilibrium-solubility limits. Increased solidification rates lead to the formation of supersaturated solid solutions and fine particles of metastable and stable intermetallic phases. The amounts of intermetallic phases are reduced and the shape is usually spherical. The slow decomposition of a supersaturated solution containing transition metals at higher temperatures can lead to the precipitation strengthening of the material. The layout of the melt-spinning process is shown in **Figure 1**.[3]

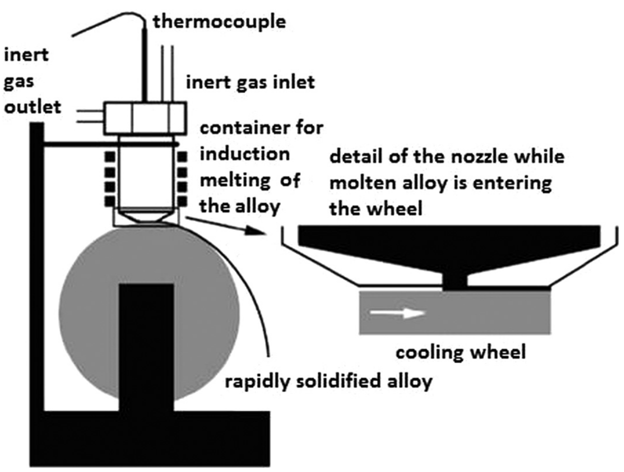

Figure 1: Melt-spinning principles
Slika 1: Shematski prikaz ulivanja na bakreni valj

To obtain a bulk material, the rapid-solidification process is associated with the compaction of the RS product. At first, the powder has to be milled, e.g., by cryogenic milling, to obtain a metallic powder with a well-preserved fine microstructure The most suitable technology is hot extrusion or e.g., hot isostatic pressing (HIP) or spark plasma sintering (SPS).[9–11]

3 EXPERIMENTAL WORK

Alloys with the chemical compositions of Al-7Fe-4Cu, Al-4Fe-4Cu-3Cr and Al-4Fe-4Cu-3Ni were prepared by melting the master alloy Al-11Fe (*w*/%) with the additions of pure Cu, Cr and Ni in an electric-resistance furnace in a graphite crucible and then poured into a brass mould. The second series was prepared by remelting the alloy and subsequent water quenching. The third series was prepared with single-roll melt spinning. The melting was carried out under an argon protective atmosphere and the temperature of the melt was 950 °C. The material was melted in a quartz-glass nozzle and then poured onto a copper wheel using overpressured argon. The circumferential speed of the wheel was 30 m s^{-1}. The process yielded aluminium-alloy ribbons approximately 40 μm thick. The metallographic cuts of the investigated alloys were etched in Kroll's reagent (10 mL HF, 5 mL HNO_3 and 85 mL H_2O) and investigated with an Olympus PME3 light microscope and a TESCAN VEGA 3 LMU scanning electron microscope (SEM) equipped with an Oxford Instruments INCA 350 EDS analyser. The phase composition was determined with X- ray diffraction (XRD, PANalytical X'Pert Pro). The mechanical properties of the investigated alloys were examined with Vickers-hardness measurements with the 5 kg (HV 5) and 0.005 kg (HV 0.005) loads. The microhardness was measured using a Neophot 2 light microscope equipped with a Hanemann microhardness tester.

4 RESULTS AND DISCUSSION

4.1 Microstructure

The microstructures of the aluminium alloys prepared by conventional casting into a brass mould are shown in **Figures 2** to **4**. It is obvious that the microstructure obtained after the conventional casting is composed of an inhomogeneous material with large amounts of coarse and brittle binary intermetallic phases $Al_{13}Fe_4$ and $CuAl_2$ and ternary phase $Al_{23}CuFe_4$ in the solid solution of the alloying elements in aluminium. Moreover, the nickel-alloyed material contains Al_4Ni_3, Al_7Cu_4Ni and $Al_{75}Ni_{10}Fe_{15}$ as well.

Figures 5 to **7** show the microstructures of the alloys prepared by melting at 1000 °C and then water quenched. Intermetallic phases $Al_{13}Fe_4$ and $CuAl_2$ become finer due

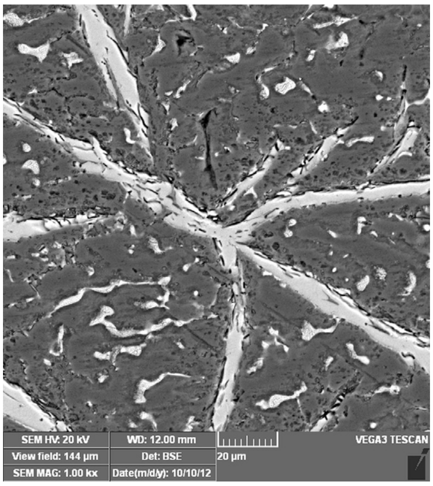

Figure 2: Microstructure of the as-cast Al-7Fe-4Cu (SEM)
Slika 2: Strjevalna struktura Al-7Fe-4Cu (SEM)

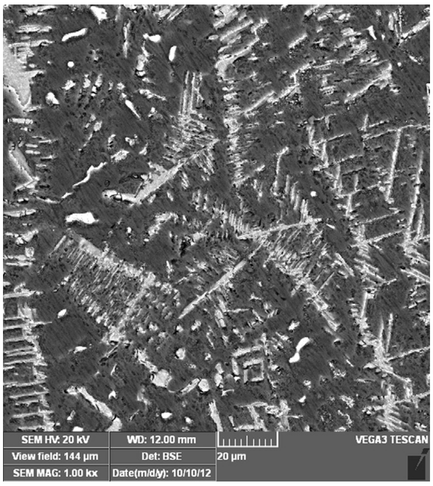

Figure 3: Microstructure of the as-cast Al-4Fe-4Cu-3Cr (SEM)
Slika 3: Strjevalna struktura Al-4Fe-4Cu-3Cr (SEM)

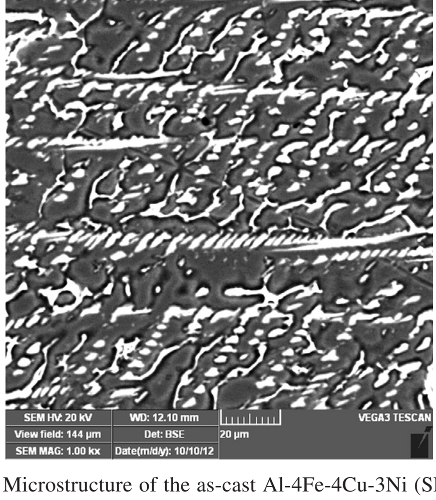

Figure 4: Microstructure of the as-cast Al-4Fe-4Cu-3Ni (SEM)
Slika 4: Strjevalna struktura Al-4Fe-4Cu-3Ni (SEM)

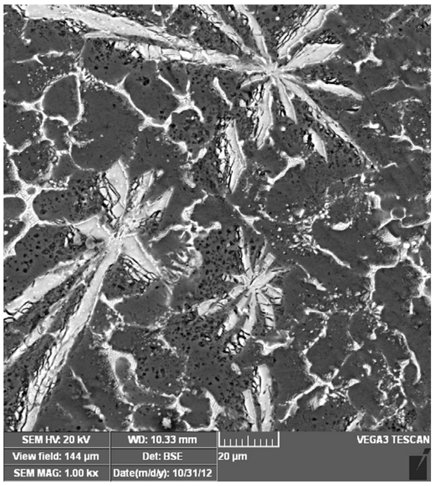

Figure 5: Microstructure of the water-quenched Al-7Fe-4Cu (SEM)
Slika 5: Mikrostruktura Al-7Fe-4Cu po ohlajanju v vodi (SEM)

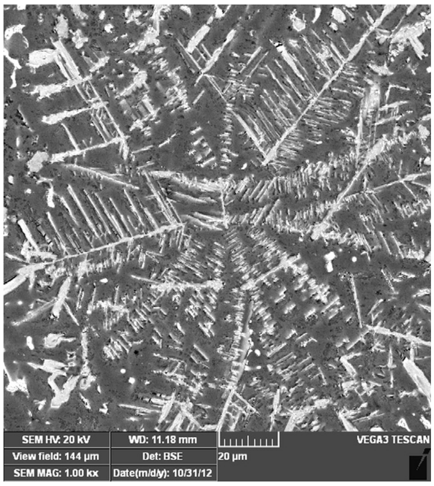

Figure 6: Microstructure of the water-quenched Al-4Fe-4Cu-3Cr (SEM)
Slika 6: Mikrostruktura Al-4Fe-4Cu-3Cr po ohlajanju v vodi (SEM)

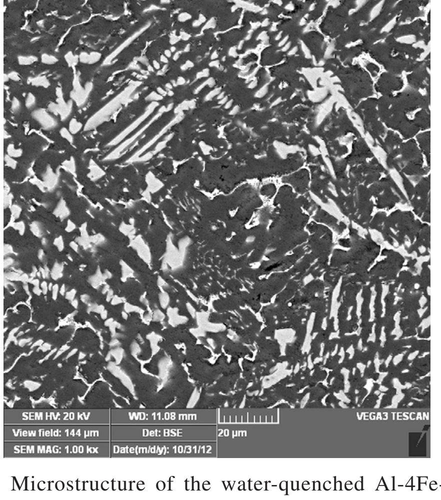

Figure 7: Microstructure of the water-quenched Al-4Fe-4Cu-3Ni (SEM)
Slika 7: Mikrostruktura Al-4Fe-4Cu-3Ni po ohlajanju v vodi (SEM)

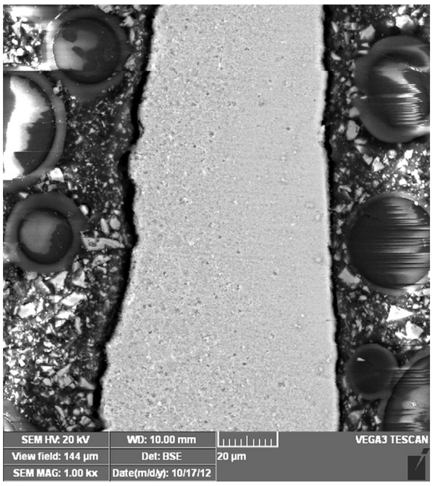

Figure 8: Microstructure of the rapidly solidified Al-7Fe-4Cu (SEM)
Slika 8: Mikrostruktura hitro strjenega traku iz Al-7Fe-4Cu (SEM)

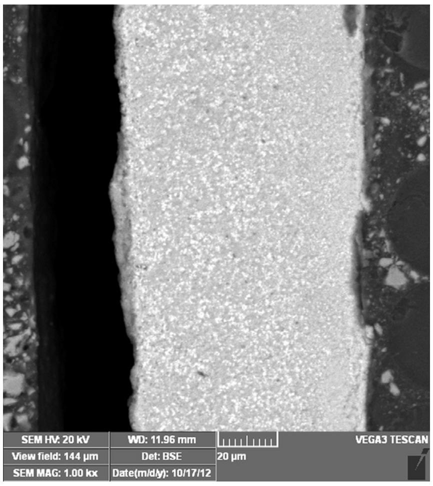

Figure 9: Microstructure of the rapidly solidified Al-4Fe-4Cu-3Cr (SEM)
Slika 9: Mikrostruktura hitro strjenega traku iz Al-4Fe-4Cu-3Cr (SEM)

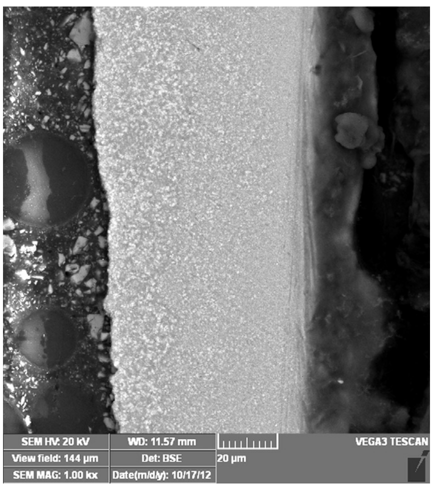

Figure 10: Microstructure of the rapidly solidified Al-4Fe-4Cu-3Ni (SEM)
Slika 10: Mikrostruktura hitro strjenega traku iz Al-4Fe-4Cu-3Ni (SEM)

to a more intensive cooling and the amount of the intermetallics in the microstructure is decreasing. In Al-7Fe-4Cu, quasicrystalline $Al_{65}Cu_{20}Fe_{15}$ is formed instead of a stable $Al_{23}CuFe_4$. On the other hand, no quasicrystalline phases were detected in the water-quenched Al-4Fe-4Cu-3Cr, but $Al_{13}Cr_2$ occurred in the microstructure. No differences in the phase composition of the alloy containing nickel in the as-cast and water-quenched states were detected.

The microstructures of the rapidly solidified alloys in the longitudinal cuts are documented in **Figures 8** to **10**. It is evident that the microstructure of a prepared ribbon is strongly dependent on the distance from the cooling wheel. On the wheel side, which is cooled more intensely, a supersaturated solid solution with nanocrystalline intermetallics is formed. On the free side, fine spherical intermetallic particles are formed. The saturation of the solution decreases when moving the ribbon from the wheel side to the free side. The amounts of the $Al_{13}Fe_4$ and $CuAl_2$ phases are negligible; instead of them, metastable phases Al_4Ni_3, $Al_{75}Ni_{10}Fe_{15}$, $Al_{23}CuFe_4$ or Al_7Cu_4Ni and quasicrystalline phase $Al_{65}Cu_{20}Fe_{15}$, are formed.[12–14] The phase compositions of all the samples

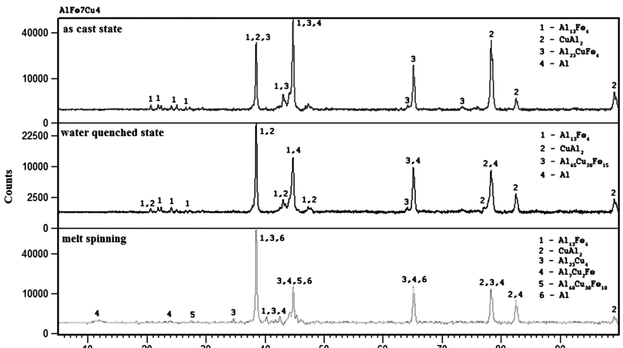

Figure 11: XRD patterns of the Al-7Fe-4Cu prepared with different methods
Slika 11: XRD-posnetki Al-7Fe-4Cu, izdelane po različnih metodah

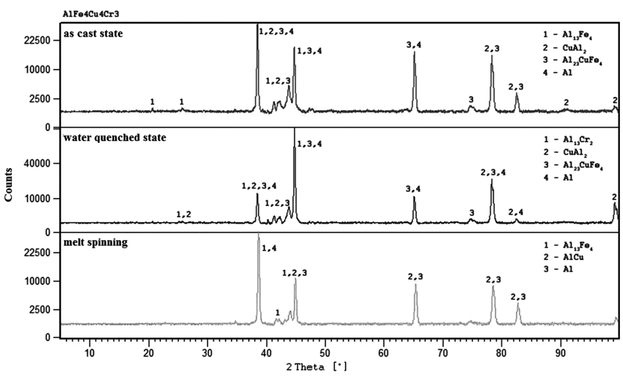

Figure 12: XRD patterns of the Al-4Fe-4Cu-3Cr prepared with different methods
Slika 12: XRD-posnetki Al-4Fe-4Cu-3Cr, izdelane po različnih metodah

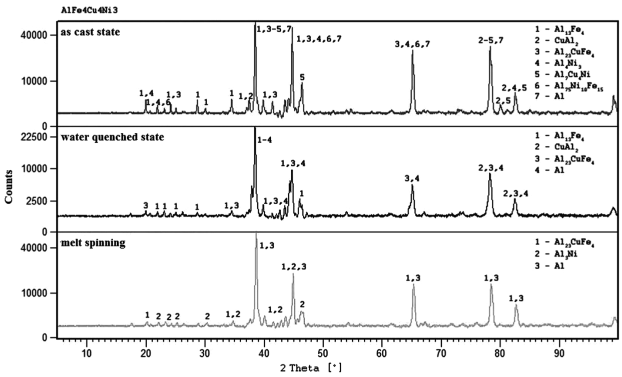

Figure 13: XRD patterns of the Al-4Fe-4Cu-3Ni prepared with different methods
Slika 13: XRD-posnetki Al-4Fe-4Cu-3Ni, izdelane po različnih metodah

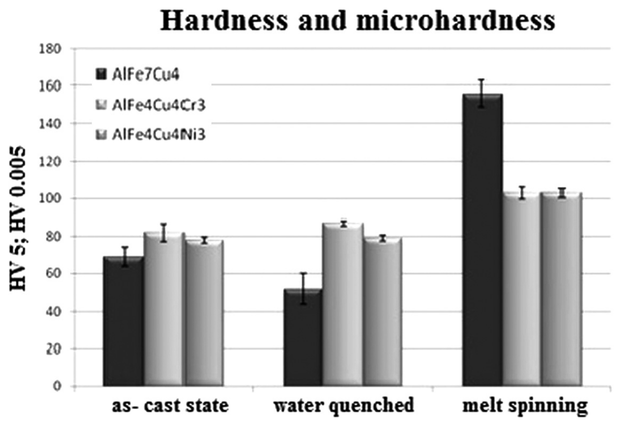

Figure 14: Hardness and microhardness measurements
Slika 14: Izmerjene trdote in mikrotrdote

are summarized in **Figures 11** to **13**. The phase composition, the amounts of intermetallics and the particle size are inevitably dependent on the solidification rate.

4.2 Hardness measurement

The Vickers hardness of the as-cast and water-quenched samples was measured with a 5 kg load. The microhardness of the rapidly solidified alloys was measured with a 5 g load because of the low thickness of the produced ribbons. The microhardness of the rapidly solidified alloys was measured in the centre of a ribbon to avoid the influence of the epoxy resin surrounding the sample. The measurement results are shown in **Figure 14**. It is obvious that the hardness increases with the increasing solidification rate. The as-cast alloys consist of large sharp-edged particles of the intermetallics that have a negative effect on the hardness. A decrease in the particle size and the strengthening caused by the presence of the supersaturated solutions are the main explanations of the increased hardness.

5 CONCLUSIONS

This work focused on a comparison of the microstructures of the Al-Cu-Fe-X alloys prepared at various solidification rates. The microstructures of the alloys prepared with traditional casting and water quenching are considerably inhomogeneous. There are large amounts of coarse $Al_{13}Fe_4$ and $CuAl_2$ intermetallic phases in the aluminium matrix. The amount of intermetallics decreases and the particles become finer, if the solidification rate increases. In the rapidly solidified alloys, the amounts of $Al_{13}Fe_4$ and $CuAl_2$ are limited because these phases are replaced by metastable and quasicrystalline intermetallics. The microstructures of RS alloys consist of aluminium supersaturated with transition metals and spherical intermetallics. The hardness of the investigated materials is hardly dependent on the cooling rate; higher values were reached for very fine materials.

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