UDK 669.715:621.7
Original scientific article/Izvirni znanstveni članek

ISSN 1580-2949
MTAEC9, 48(3)349(2014)

MICROSTRUCTURE OF RAPIDLY SOLIDIFIED AND HOT-PRESSED Al-Fe-X ALLOYS

MIKROSTRUKTURA HITRO STRJENIH IN VROČE STISKANIH ZLITIN Al-Fe-X

Milena Voděrová, Pavel Novák, Dalibor Vojtěch

Institute of Chemical Technology, Department of Metals and Corrosion Engineering, Technická 5, 166 28 Prague 6, Czech Republic
voderovm@vscht.cz

Prejem rokopisa – received: 2013-06-07; sprejem za objavo – accepted for publication: 2013-08-28

Rapidly solidified aluminium alloys are promising materials for many technical applications. The main advantages of aluminium alloys alloyed with transition metals, such as Ni, Fe, Cr or Mn, are a good strength-to-weight ratio, a relatively low price, improved mechanical properties and a thermal stability. Mechanical properties of these alloys can be improved with rapid-solidification techniques. In this work, a production method employing melt spinning, cryogenic milling of melt-spun ribbons and the subsequent hot pressing was tested. The Al-11Fe, Al-7Fe-4Cr and Al-7Fe-4Ni alloys were processed in this way. The microstructure and mechanical properties of the prepared alloys were compared with the cast materials of the same composition. The results showed that the proposed technology can lead to a significant microstructure refinement and hardness improvement of the investigated alloys, when compared with the as-cast state. In the case of the Al-Fe alloy, a local coarsening of the $Al_{13}Fe_4$ particles was observed. Hot pressing is suggested to be a promising method for processing rapidly solidified alloys with respect to maintaining a fine structure and satisfactory hardness.

Keywords: rapid solidification, aluminium alloys, hot pressing, microstructure

Hitro strjene aluminijeve zlitine so obetajoč material za različno tehnično uporabo. Glavne prednosti aluminijevih zlitin, legiranih s prehodnimi kovinami, kot so Ni, Fe, Cr ali Mn, so ugodno razmerje med trdnostjo in maso, relativno nizka cena, izboljšane mehanske lastnosti in termična stabilnost. Mehanske lastnosti teh zlitin se lahko izboljša s tehniko hitrega strjevanja. Preizkušena je bila metoda izdelave z uporabo ulivanja tankega traku, kriogeno mletje tankega ulitega traku in končno vroče stiskanje prahu. Tako so bile izdelane zlitine Al-11Fe, Al-7Fe-4Cr in Al-7Fe-4Ni. Mikrostruktura in mehanske lastnosti izdelanih zlitin so bile primerjane z litim materialom z enako sestavo. Rezultati so pokazali, da predložena tehnologija, v primerjavi z litim stanjem, povzroča občutno drobnejšo mikrostrukturo in povišanje trdote. Pri zlitini Al-Fe je bilo opaženo lokalno povečanje delcev $Al_{13}Fe_4$. Domneva se, da je vroče stiskanje obetajoča metoda za obdržanje drobnozrnate strukture in zadovoljive trdote.

Ključne besede: hitro strjevanje, aluminijeve zlitine, vroče stiskanje, mikrostruktura

1 INTRODUCTION

Rapidly solidified aluminium alloys alloyed with transition metals are promising materials for the technical applications requiring improved mechanical properties and a thermal stability.[1,2] However, to be applicable, the powders obtained with atomisation or the thin ribbons obtained with melt spinning have to be compacted. To produce a dense material, several methods are recommended: hot isostatic pressing (HIP), hot extrusion, spark-plasma sintering (SPS) or hot pressing (HP).[3–6] The main request about the manufacturing method for producing a bulk material is to keep the particle size at the lowest possible level. All the listed methods require relatively high temperatures and pressures. Although HIP is beneficial in engineering, it is quite complicated for a laboratory use because of the complex equipment required. For the aluminium alloys investigated in this paper, hot extrusion or hot pressing are the most suitable compaction technologies. Recently, the extrusion of rapidly solidified aluminium alloys has been widely investigated.[7,8] However, in the case of the alloys containing high amounts of intermetallics, such as the investigated Al-Fe-X alloys, hot extrusion requires high temperatures and pressures. In this work, hot pressing as a less demanding alternative was tested. In recent years, the consumption of aluminium alloys in engineering has been rising and this is closely connected with the issue of a waste disposal. Aluminium scrap is often contaminated with a mixture of the elements coming from the steel parts, e.g., Fe, Ni, Cr, Mn, that are very difficult and costly to remove. However, these elements are often recommended to improve the thermal stability of aluminium alloys. The idea that powder metallurgy can be used to recycle the aluminium contaminated with a mixture of elements was already mentioned in[7]. The alloys used in this experiment serve as a model leading to the development of a process for recycling aluminium scrap containing iron and stainless steel.

2 EXPERIMENTAL WORK

The samples of the Al-11Fe, Al-7Fe-4Ni and Al-7Fe-4Cr (mass fractions, *w*/%) alloys were prepared by conventional casting, melt spinning and hot pressing. The chemical compositions of the investigated alloys were chosen in order to model the metallic waste containing, e.g., the stainless steel including relatively high amounts of nickel and chromium. The alloys were prepared by melting the Al-11Fe master alloy and the master alloy with an addition of pure nickel or chromium. In the first step, the alloys were melted in an electric-resistance furnace at 1000 °C and cast into a non-preheated brass mould. After that, the alloys were subjected to a rapid solidification with the melt-spinning technique. In this process, a molten alloy was cast onto a copper-alloy wheel. The melting was carried out under argon protective atmosphere; the temperature of the melt was 1200 °C because of the high liquidus temperatures of the used alloys. The process yields aluminium-alloy ribbons, 30 μm thick. The rotation velocity of the cooling wheel in the melt-spinning process was 1420 r/min, which corresponds to the circumferential speed of 38 m/s. The rapidly solidified alloys were cryogenically milled in a planetary ball mill RETSCH PM 100 in liquid nitrogen in order to ensure an embrittlement of the ribbons and to simplify the milling. The milling was performed for 10 min at 400 r/min. For compacting the material, a universal testing machine LabTest5.250SP1 was used. The milled powders were cold pre-pressed under the pressure of 220 MPa and then heated at 500 °C for 20 min to ensure temperature homogeneity. Hot pressing was performed for 5 min at 500 °C with the maximum pressure of 530 MPa. After hot pressing, a sample was cooled on air. The microstructures of all the samples were investigated with a TESCAN VEGA 3 LMU scanning electron microscope (SEM) equipped with an Oxford Instruments INCA 350 EDS analyser. The mechanical properties of the investigated alloys were examined by measuring the Vickers hardness and microhardness with the 5 kg (HV 5) and 0.005 kg (HV 0.005) loads at room temperature. The results are presented in the form of the average of ten values. The phase composition was determined with X- ray diffraction (XRD, PANalytical X´Pert Pro).

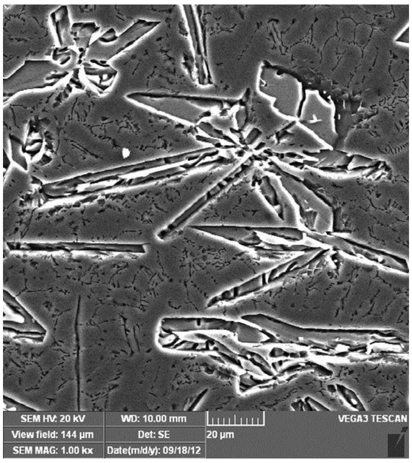

Figure 1: Microstructure of as-cast Al-11Fe (SEM)
Slika 1: Mikrostruktura litega Al-11Fe (SEM)

3 RESULTS AND DISCUSSION

3.1 Microstructure

The microstructures of the as-cast alloys obtained with the scanning electron microscope are shown in the figures, acquired in the backscattered electron mode (BSE). A slow solidification rate causes the evolution of large amounts of irregularly shaped, coarse intermetallic phases. As seen in **Figure 1**, the conventionally cast Al-11Fe consists of large grains of a solid solution of Fe in Al and coarse $Al_{13}Fe_4$ intermetallics (also referred to as $FeAl_3$ in[9]). The nickel added to the as-cast alloy (**Figure 2**) remains dissolved together with the iron in aluminium, while the iron forms coarse particles of $Al_{13}Fe_4$. In an as-cast alloy, chromium is partly dissolved in aluminium or it forms Al_5Cr and $Al_{13}Cr_2$ intermetallics together with stable $Al_{13}Fe_4$, as seen in the **Figure 3**. After increasing the solidification rate, the microstructures of the investigated alloys change significantly. The microstructure of the rapidly solidified ribbon in a longitudinal cut is composed of two main areas; the wheel side and the free side. The wheel side is in the direct contact with

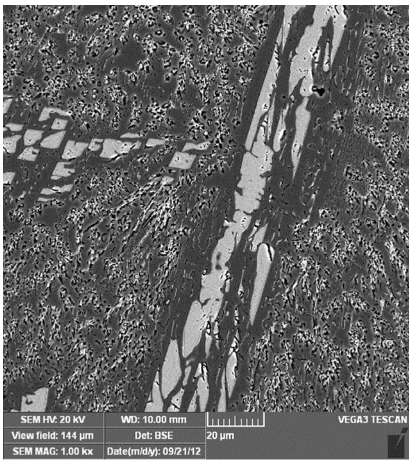

Figure 2: Microstructure of as-cast Al-7Fe-4Ni (SEM)
Slika 2: Mikrostruktura litega Al-7Fe-4Ni (SEM)

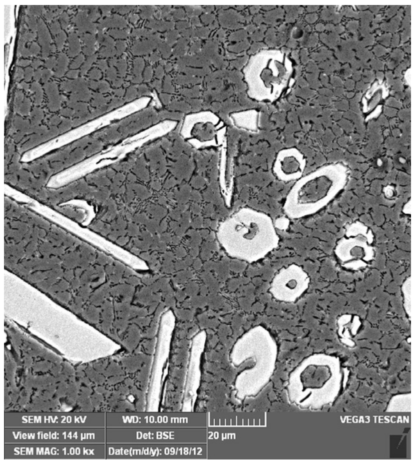

Figure 3: Microstructure of as-cast Al-7Fe-4Cr (SEM)
Slika 3: Mikrostruktura litega Al-7Fe-4Cr (SEM)

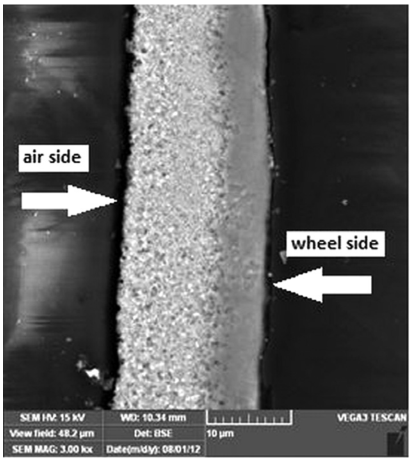

Figure 4: Microstructure of melt-spun Al-11Fe (SEM)
Slika 4: Mikrostruktura hitro strjenega traku iz Al-11Fe (SEM)

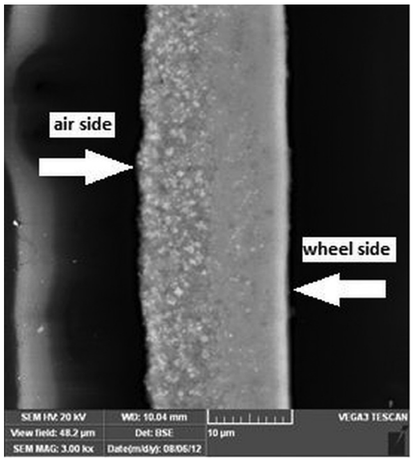

Figure 5: Microstructure of melt-spun Al-7Fe-4Ni (SEM)
Slika 5: Mikrostruktura hitro strjenega traku iz Al-7Fe-4Ni (SEM)

the cooling wheel and it is usually composed of a supersaturated solid solution of the alloying elements in aluminium and low amounts of stable and metastable intermetallics and/or quasicrystals. The free side is cooled less intensely. This area contains more intermetallics that are very fine and round-shaped. The microstructure of the rapidly solidified Al-11Fe alloy in **Figure 4** is composed of a supersaturated solid solution of Fe in Al and nanometre-sized intermetallic phases on the wheel side, while the stable $Al_{13}Fe_4$ and metastable Al_6Fe phases are located on the free side. Both regions of the RS Al-Fe-Ni alloy are composed of a supersaturated solid solution of Ni and Fe in Al, quasicrystalline $Al_{75}Ni_{10}Fe_{15}$ and stable $Al_{13}Fe_4$ and Al_3Ni_2 phases (**Figure 5**). The microstructure of the melt-spun alloy containing chromium in **Figure 6** is composed of a supersaturated solid solution, $Al_{13}Cr_2$ and $Al_{13}Fe_4$ on both sides of the ribbon.

3.2 Milling of rapidly solidified alloys

Rapidly solidified alloys were milled in liquid nitrogen to ensure the embrittlement of the ribbons. The optimum time for milling was determined as 10 min. After 5 min of milling, there was still a large amount of unmilled ribbons, while after 15 min all the nitrogen evaporated; the product became very hot, forming clusters instead of the required powder. The microstructure of milled ribbons is shown in **Figure 7**. From these micrographs it can be seen that the cryogenic milling of RS ribbons does not produce round, but irregularly shaped porous particles of various sizes.

3.3 Hot pressing of rapidly solidified milled powders

The microstructures of the hot-pressed alloys are shown in **Figures 8** to **10**. It is obvious that the microstructure of the hot-pressed Al-11Fe coarsened significantly. The metastable Al_6Fe and supersaturated solution

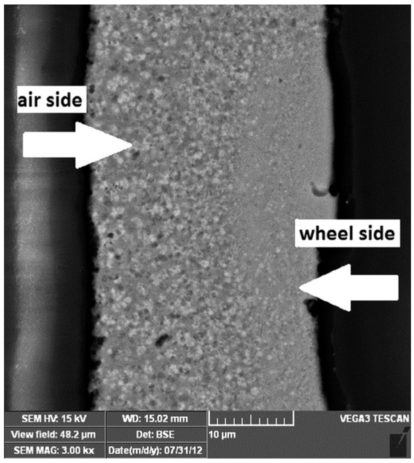

Figure 6: Microstructure of melt-spun Al-7Fe-4Cr (SEM)
Slika 6: Mikrostruktura hitro strjenega traku iz Al-7Fe-4Cr (SEM)

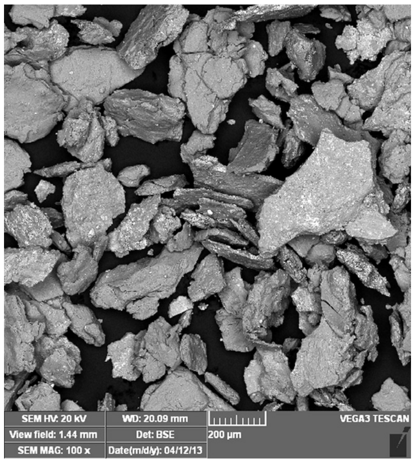

Figure 7: Al-11Fe powder obtained with cryogenic milling (SEM)
Slika 7: Al-11Fe-prah, dobljen s kriogenim mletjem (SEM)

were decomposed to form a large amount of $Al_{13}Fe_4$. Al-7Fe-4Ni has a fine microstructure including fine particles of $Al_{13}Fe_4$ and Al_4Ni_3. The microstructure of the hot-pressed Al-7Fe-4Cr does not change significantly. It contains fine, homogeneously distributed particles of the solid solution, and the $Al_{13}Fe_4$ and $Al_{13}Cr_2$ intermetallic phases. These results suggest a better thermal stability of the ternary alloys. The phase compositions of the investigated alloys in the as-cast, rapidly solidified and hot-pressed state are compared in **Figures 11** to **13**. In the case of Al-11Fe, the as-cast alloy contains a high amount of $Al_{13}Fe_4$ which is transformed, due to a lack of diffusion time, into Al_6Fe after rapid solidification. This metastable phase is then decomposed during hot pressing and a high fraction of $Al_{13}Fe_4$ is formed. The quasicrystals of $Al_{75}Ni_{10}Fe_{15}$ present in the rapidly solidified Al-7Fe-4Ni decompose after hot pressing. The phase

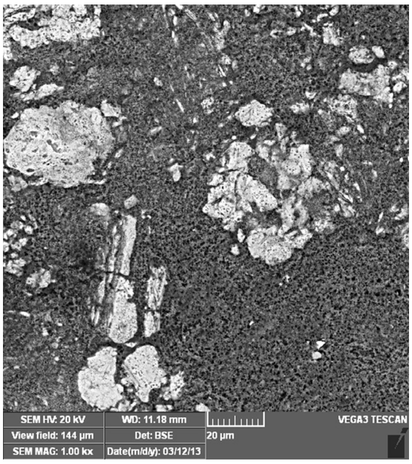

Figure 8: Microstructure of hot-pressed Al-11Fe (SEM)
Slika 8: Mikrostruktura vroče stisnjene zlitine Al-11Fe (SEM)

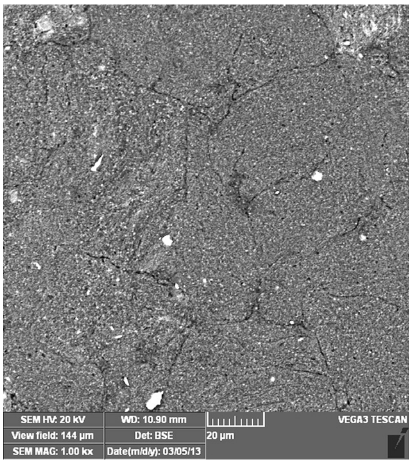

Figure 9: Microstructure of hot-pressed Al-7Fe-4Ni (SEM)
Slika 9: Mikrostruktura vroče stisnjene zlitine Al-7Fe-4Ni (SEM)

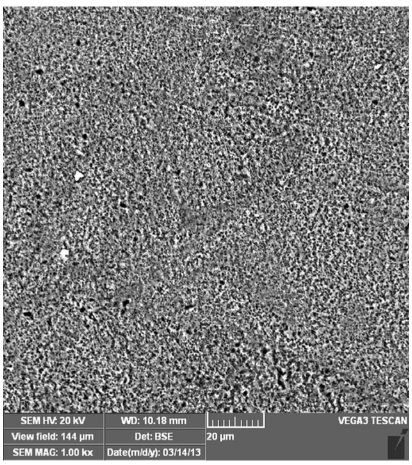

Figure 10: Microstructure of hot-pressed Al-7Fe-4Cr (SEM)
Slika 10: Mikrostruktura vroče stisnjene zlitine Al-7Fe-4Cr (SEM)

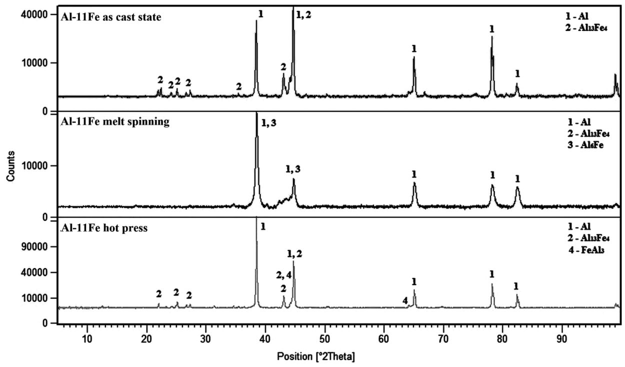

Figure 11: XRD patterns of Al-11Fe prepared with different methods
Slika 11: XRD-posnetki zlitine Al-11Fe, pripravljene z različnimi metodami

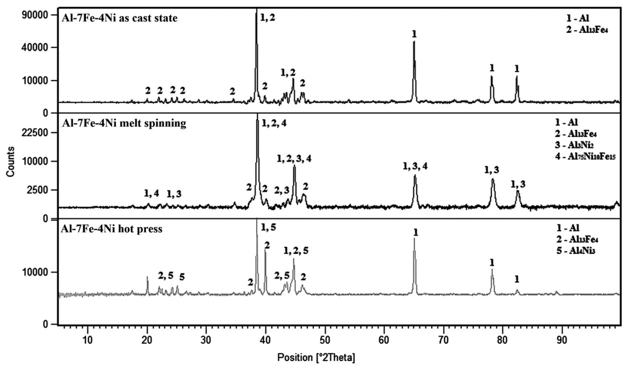

Figure 12: XRD patterns of Al-7Fe-4Ni prepared with different methods
Slika 12: XRD-posnetki zlitine Al-7Fe-4Ni, pripravljene z različnimi metodami

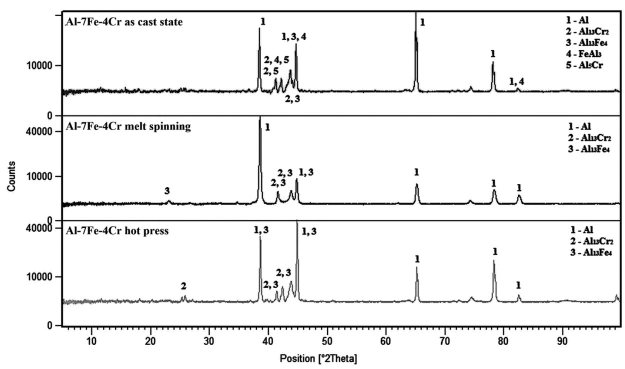

Figure 13: XRD patterns of Al-7Fe-4Cr prepared with different methods
Slika 13: XRD-posnetki zlitine Al-7Fe-4Cr, pripravljene z različnimi metodami

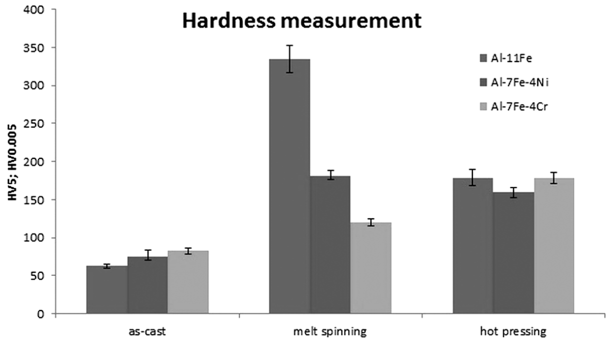

Figure 14: Hardness measurement
Slika 14: Izmerjene trdote

composition of Al-7Fe-4Cr changes after melt spinning but remains the same after further processing.

3.4 Hardness measurement

The results obtained with the hardness and microhardness measurements are summarized in **Figure 14**. The as-cast and hot-pressed materials were measured with the load of 5 kg, while the RS ribbons were measured with the load of 0.005 kg. The microhardness measurement was realised in the centre of the ribbons to avoid interference between the material and the epoxy surrounding the samples. From the diagram it is evident that the increasing solidification rate, resulting in a refined microstructure, increased the hardness as well. A significantly higher value of Al-11Fe is probably caused by a large amount of the hard metastable Al_6Fe. On the other hand, the hardness of Al-11Fe and Al-Fe-Ni decreased after hot pressing, while the hardness of Al-Fe-Cr increased. The decrease in the hardness can be explained as the result of intermetallic coarsening, a decomposition of the metastable Al_6Fe in Al-11Fe and a decomposition of the quasicrystals in Al-7Fe-4Ni. On the contrary, the $Al_{13}Cr_2$ formed in the rapidly solidified Al-7Fe-4Cr is probably more stable and its hardening effect remains present even after hot pressing. In addition, chromium has a lower diffusivity in aluminium than iron and nickel, thus blocking the coalescence and recrystallization of the phases.

However, the elevated temperatures caused a moderate decrease in the hardness. A significant change in the hardness occurred in the case of Al-Fe-Cr, where the values for the rapidly solidified samples increased up to 180 HV, probably due to a precipitation of the $Al_{13}Cr_2$ intermetallic phase.

4 CONCLUSION

In this work, the processing of Al-Fe-X alloys with powder-metallurgy technology including melt spinning and hot pressing was tested. Traditionally, cast alloys are composed of an aluminium matrix containing partly dissolved aluminium elements and coarse intermetallics such as $Al_{13}Fe_4$ in the case of Al-11Fe and Al-7Fe-4Ni, and $Al_{13}Cr_2$ and Al_5Cr in Al-7Fe-4Cr. The rapid-solidification results are given for fine-grained microstructures consisting of supersaturated solid solutions, stable and metastable intermetallic phases. In Al-11Fe, a supersaturated solid solution and metastable phase Al_6Fe are formed, which is probably the main reason for a higher microhardness of the melt-spun ribbons. The melt-spun Al-7Fe-4Ni alloy contains Al_3Ni_2 and a quasicrystalline phase ($Al_{70}Ni_{10}Fe_{15}$). The microstructure of the RS chromium alloy differs only in the amount of intermetallics, which is decreased due to the suppressed diffusion. Rapidly solidified alloys reach the highest values of microhardness primarily due to a very fine microstructure. The microstructures of hot-pressed alloys are fine and homogeneous, except for Al-11Fe where the microstructure coarsened significantly, presumably because of a decomposition of metastable Al_6Fe to the more stable $Al_{13}Fe_4$. This is also the reason for a rapid decrease in the microhardness, while Al-7Fe-4Ni loses only about 10 HV and the microhardness of Al-7Fe-4Cr is even increased. This is probably caused by a large amount of chromium aluminides that did not decompose during milling and hot pressing. All the results show that these alloys can be beneficial in technical engineering and suggest that the processing methods are applicable. The thermal stability of these alloys and mechanical properties at elevated temperatures will be studied further.

Acknowledgement

This research was financially supported by the Czech Science Foundation, project No. P108/12/G043.

5 REFERENCES

[1] E. J. Lavernia, T. S. Srivatsan, Journal of Materials Science and Technology, 45 (**2010**), 287–325
[2] P. Jurči, M. Dománková, M. Hudáková, B. Šuštaršič, Mater. Tehnol., 41 (**2007**) 6, 283–287
[3] S. Billard, J. P. Fondere, B. Bacroix, G. F. Dirras, Acta Materialia, 54 (**2006**), 411–421
[4] N. L. Loh, K. Y. Sia, Journal of Materials Processing Technology, 30 (**1992**), 45–65
[5] L. Wang, J. Zhang, W. Jiang, Int. Journal of Refractory Metals and Hard Materials, 39 (**2013**), 103–112
[6] F. Vollertsen, A. Sprenger, J. Kraus, H. Arnet, Journal of Materials Processing Technology, 87 (**1999**), 1–27
[7] F. Průša, D. Vojtěch, Materials Science and Engineering, A, 565 (**2013**), 13–20
[8] D. Vojtěch, A. Michalcová, F. Průša, K. Dám, P. Šedá, Materials Characterization, 56 (**2012**), 83–97
[9] C. Chen, J. Wang, D. Shu, P. Li, J. Xue, B. Sun, Materials Transactions, 52 (**2011**), 1629–1633