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THE STRUCTURE AND PROPERTIES OF WROUGHT ALUMINIUM ALLOYS SERIES 6XXX WITH VANADIUM FOR AUTOMOTIVE INDUSTRY "

Marzena Lech-Grega, PhD, Eng., IMN Prof. mlechgrega@imn.skawina.pl
Co-Authors: W.Szymański, PhD, Eng., B.Płonka, PhD, Eng., S.Boczkal, PhD, Eng., M.Gawlik, MSc, Eng, M.Bigaj, MSc, Eng., P.Korczak, MSc, Eng.
Institute of Non-Ferrous Metals in Gliwice, Light Metals Division in Skawina, Pilsudskiego 19, 32-050 Skawina, Poland.
www.imn.skawina.pl

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Abstract

The increasing number of vehicle parts, including elements operating in crumple zones, are manufactured from light metal alloys based on aluminum and magnesium. The choice of material for these applications should consider not only the mechanical properties of the alloy, but also its ability to absorb energy.

The materials that can offer these properties are wrought aluminum alloys containing vanadium in an amount of 0.05 - 0.5%, characterized by high strength and high toughness. These alloys are also characterized by an adequate capacity to absorb the kinetic energy (low value of the $R_{p0.2}/R_m$ ratio and very high reduction of area Z).

The paper describes structure examinations and testing of mechanical properties using directly extruded round and square bars and forgings made from ingots and bars. The examined material was aluminum alloy from the 6xxx series containing 0.1, 0.2 and 0.3% vanadium.

The extruded profiles were characterized by the mechanical properties comparable with the strength parameters of profiles made from a similar alloy from the 6xxx series but without the addition of vanadium, except for the elongation that assumed much higher values. The use of water wave on the press prevented the formation of a rim of coarse crystals on the profile cross-section.

Introduction

In connection with the EU directives on energy efficiency, fuels included, solutions are looked for to reduce energy consumption by reducing the weight of, among others, transport means. The increasing number of vehicles (cars, trains, motorcycles) are made from light metal alloys and, more exactly, from aluminum and magnesium alloys.

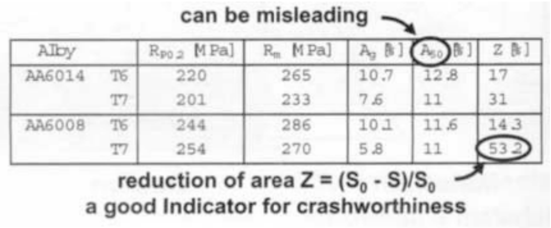
The materials from which these vehicles are manufactured are characterized by completely different properties, depending on the application, that is, on the type of the fabricated element.

Aluminum alloys are the lightweight materials whose physical characteristics can be controlled by the chemical composition and the processing route, using either the traditional methods like casting or plastic forming (rolling, extrusion, forging) or completely new processes like rapid solidification, tixoforming or production of foams.

For the safety of vehicles, a new group of wrought aluminum alloys containing vanadium in the range of 0.05 - 0.5% is currently developed, mainly to obtain high mechanical properties combined with good toughness and, additionally, to ensure the stability of properties at elevated temperatures. European standards specify alloys from the 6xxx series, such as EN-AW 6260, EN-AW 6008 and EN-AW 6014, all with additions of up to 0.2% vanadium.

Production of these alloys is covered by various European and U.S. patents, but each of these patents stresses the importance of various alloying additions and their percent content in alloy.

The above mentioned alloys are characterized by the ability to absorb kinetic energy [1], a characteristic very important in these materials, especially when used for parts operating in the crumple zone of cars during accidents (low $R_{p0,2}/R_m$ ratio and very large reduction of area Z).



can be misleading

Alloy		$R_{p0.2}$ [MPa]	R_m [MPa]	A_g [%]	A_{50} [%]	Z [%]
AA6014	T6	220	265	10.7	12.8	17
	T7	201	233	7.6	11	31
AA6008	T6	244	286	10.1	11.6	14.3
	T7	254	270	5.8	11	53.2

reduction of area $Z = (S_0 - S)/S_0$
a good Indicator for crashworthiness

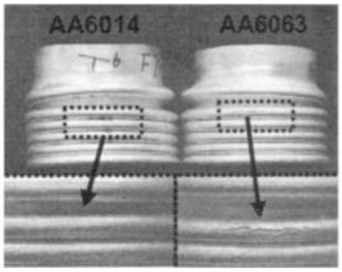


Fig. 1. Properties of vanadium-containing alloy and a comparison of alloys 6063 and 6014 after „crash test" [1].

The role of vanadium in aluminum alloys, particularly for a low content of this element, is still not known, and data available in the published literature are very scarce.

It is generally believed that vanadium acts as a grain refiner, and also reduces the conductivity and increases the temperature of recrystallization. The investigations carried out on the 6063 alloy with 0.1% vanadium have proved that vanadium increases the kinetics of the precipitation of the magnesium-silicon β" and β' phases, which in turn affects the tensile and yield strengths of the material in the T6 condition [8].

From the Al-V phase equilibrium diagram it follows that vanadium solubility in aluminum is 0.37 wt% at the temperature of 660^0C, and up to the content of about 9% vanadium, the $Al_{21}V_2$ phase can exist in the alloy[9].

Test material

Tests were carried out on extruded alloys from the 6xxx series with additions of vanadium in the amount of 0.1, 0.2 and 0.3 wt%. From these alloys, ingots of ϕ 100mm were cast by a semi-continuous process (DC Direct Cast). The ingots were next homogenized and extruded into a 20mm diameter round bar, a square bar of 20 mm side, and a hollow square profile with a square hole.

The direct extrusion tests were carried out on a horizontal press of maximum 500T capacity (Fig. 2). Before extrusion, the material was subjected to zone heating in an induction heater, which resulted in that one end of the ingot was preheated to a temperature higher by about 10°C than the opposite end. The aim was to obtain quasi-isostatic conditions in the direct extrusion process, or a uniform temperature distribution on the entire length of the extruded bar. The direct extrusion was carried out in a temperature range of 520°C - 510°C at an initial ram speed of 0.7 mm/s. When the extruded bar was caught in a puller, the ram speed (Vt) was increased in a range of 1.5 to 5 mm /s. In all tests, the press recipient was preheated to a temperature of 490°C.

Fig. 2. A horizontal direct-indirect press of maximum 500T capacity.

Test methods

Structure was examined under an Olympus GX71 light microscope with etching of phases that occurred in alloys and revealing the grains by Barker method for the examinations under polarized light.

Chemical composition of the precipitates traced in the examined alloys was identified by EDS (Energy Dispersive Spectroscopy) using Philips XL30 SEM and high-resolution S/TEM Tecnai G2 20 200kV TEM with STEM. Phase analysis was performed by XRD (X-Ray Diffraction) using Bruker D8 Advance X-ray diffractometer. Additionally, by EBSD technique (Electron Backscatter Diffraction), crystallographic parameters of phases were determined with an analysis of their orientation.

The mechanical properties of the tested materials were determined in a static tensile test and compression test using Instron DX600 600kN and 100kN 5585 testing machines, and measuring the HB and HV hardness with Duramin 2500E hardness tester and Buehler Micromet 5103 microhardness tester.

Structure of ingots

As a vanadium-based master alloy for ingots cast by the semi-continuous method (DC Direct Cast), an AlV5 alloy was used; its structure is shown in Fig 3. The X-ray phase analysis (XRD) and SEM EBSD analysis (Fig. 4) have proved that in the AlV5 master alloy the intermetallic phase containing vanadium is Al_3V. It is a monoclinic phase with Pearson symbol tI8, space group I4/mmm and lattice parameters a = 0.3780 and c = 0.8321 [2].

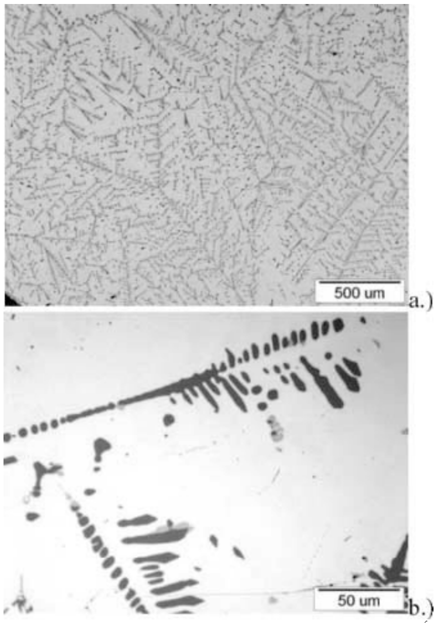


Fig. 3 Microstructure of AlV5 alloy - a.) – 50x, b.) – 500x.

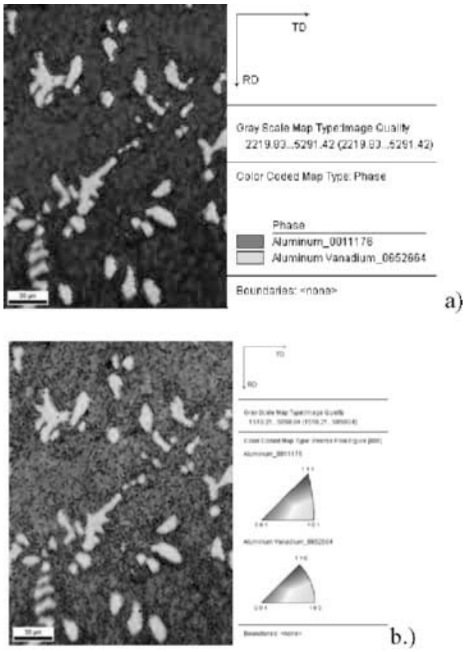


Fig.4. Mapping of phases distribution a.) and of crystallographic orientation b.) performed by EBSD method on sample of the AlV5 vanadium master alloy [2].

The cast ingots with an addition of 0.1 wt% vanadium (RV10), 0.2 wt% vanadium (RV20) and 0.3 wt% vanadium (RV30) were characterized by a homogeneous and fine-grain structure on the entire ingot cross-section. Vanadium addition to base alloy reduced the grain size [3,4] (Table 1), which proves that this element can act as a grain refiner.

Table 1 Average grain size compared for ingots RV0, RV10, RV20 and RV30

Ingot symbol	**Place of measurement**			**Mean value [µm]**
	ingot edge [µm]	**ingot half-radius [µm]**	**ingot center [µm]**	
RV0	86	111	116	104
RV10	82	94	103	93
RV20	78	90	114	94
RV30	77	90	107	91

The studies of microstructure revealed the presence of precipitates typically encountered in the 6xxx series alloys, i.e. an AlFeSi intermetallic iron phase containing vanadium - $Al_{17}(Fe_{3,2}Mn_{0,8})Si_2$ (Fig. 7) and AlFe phase – Al_3Fe, both identified in earlier work [5], as well as the strengthening Mg_2Si phase. Additionally, 0.3 wt% vanadium alloys contained the secondary angular precipitates of vanadium identified by XRD and EBSD as $Al_{18}Mg_3V_2$ (space group Fd3m, lattice parameter a=b=c=14.61Å) (Figs. 5 and 6) [2].

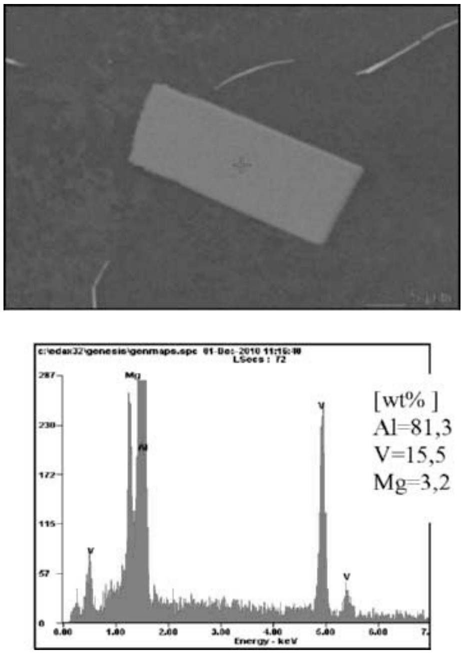


Fig.5. The precipitates of AlMgV as seen under SEM with EDS analysis (ingot RV20).

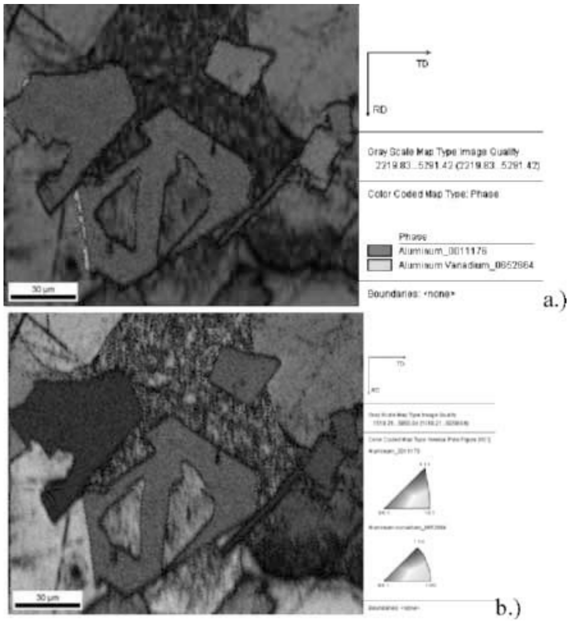


Fig. 6. Mapping of phases distribution a.) and of crystallographic orientation b.) done by EBSD on sample of the ingot RV30[2].

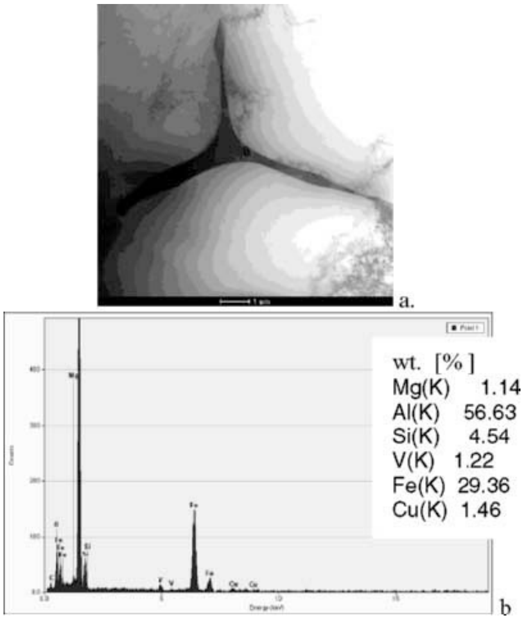


Fig.7. Iiron phase containing vanadium as seen under TEM with EDS chemical analysis.

The number of large angular precipitates of the $Al_{18}Mg_3V_2$ phase observed in ingots was directly proportional to the vanadium content in alloy, and inversely proportional to the solidification rate, and thus to the ingot diameter.

The homogenizing treatment had practically no effect on the morphology of large, block-shaped, vanadium precipitates, although chemical analysis showed a decrease in the percent content of vanadium in these precipitates (from 15 to 12%) [6].

In alloy containing 0.2% vanadium, the homogenizing treatment caused a much more serious drop in the mechanical properties of the ingots, in which vanadium rested almost entirely in the matrix. Since in the ingots containing 0.3 wt% vanadium, angular precipitates of the $Al_{18}Mg_3V_2$ phase occurred, the idea of subjecting these ingots to plastic forming by extrusion was finally given up.

Properties of round and square bars [7]

For the vanadium content of 0.1 and 0.2 wt%, the macrostructure of extruded bars was similar in both round and square cross-sections, and in either case it was characterized by the presence of a relatively wide (about 3 mm) rim of the coarse crystals (Fig. 8).

For given vanadium content levels, microstructures on the cross-sections and longitudinal sections of the round and square bars were similar because of the similar extrusion ratio λ. The alloy with 0.2% vanadium contained single angular precipitates of the $V_2Mg_3Al_{18}$ phase of the size of up to 50 μm, the morphology of which did not change during plastic forming. The precipitates of the Mg_2Si phase and of iron phases were evenly distributed within the entire volume of the profiles.

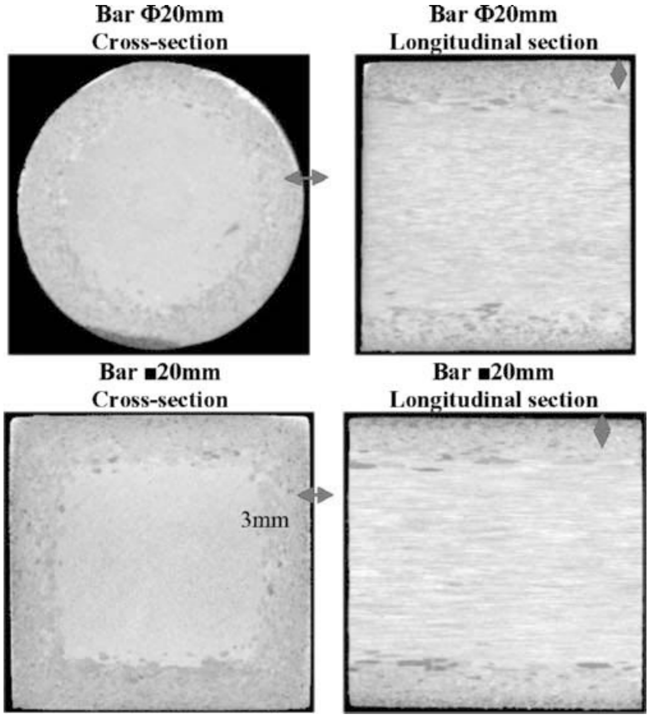


Fig.8 Macrostructure of round and square bars extruded from the 6xxx alloy with an addition of vanadium.

The average size of the very fine grains in the central part of the round and square bars was similar (about 15μm), but in the bar with square cross-section, the standard grain size deviation doubled, thus showing a large scatter in the grain size values.

In the alloy matrix, besides the strengthening Mg_2Si phase, fine-dispersed, iron-vanadium precipitates identified by EDS on TEM also occurred. Their presence had a decisive influence on alloy properties after the heat treatment (Fig. 9).

This was reflected in the hardness measurements (Fig. 10). The hardness of the round bar was higher than that of the square bar. The hardness of the alloy containing 0.2% vanadium was higher than the hardness of the alloy containing 0.1% V.

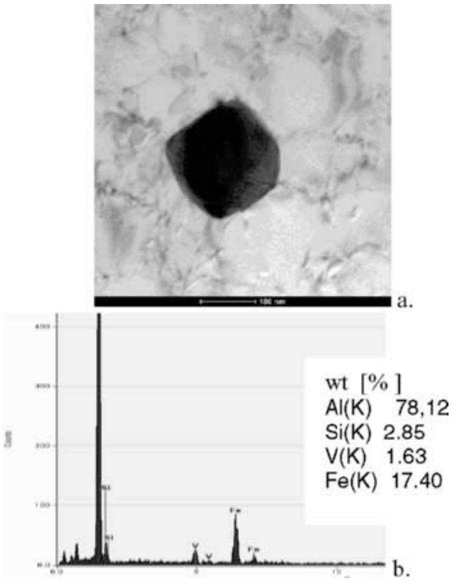


Fig.9. .Fine-dispersed vanadium precipitates as seen under TEM with EDS chemical analysis.

The mechanical properties are shown in Table 2.

Table 2 Mechanical properties of round and square bars made from the 6xxx alloy with an addition of 0.1 and 0.2 wt% vanadium; O – round bar, K –square bar.

Extruded solid profiles						
Sample	**$R_{p0.2}$ [MPa]**	**R_m [MPa]**	**A [%]**	**$E_{Rp0.2}$ [J]**	**E_{Rm} [J]**	**E_{pek} [J]**
RV10– O	94	211	21,8	1,1	78	115
RV10– K	90	229	21,0	0,9	90	127
RV20– O	102	225	21,3	1,2	89	123
RV20– K	102	223	20,4	1,5	88	120

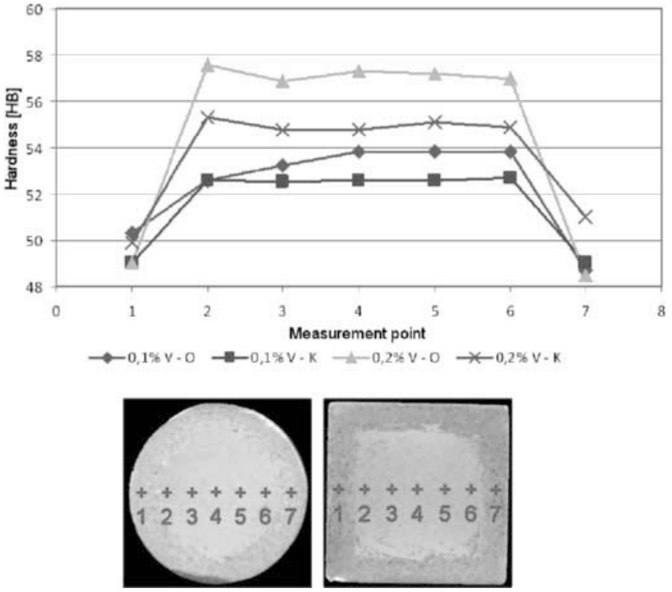


Fig. 10 Graph showing changes in hardness on the cross-sections of round and square bars made from the 6xxx alloy with an addition of 0.1 and 0.2 wt% vanadium; O - round bar, K – square bar.

The extruded rods had similar properties, but the, observed in hardness measurements, tendency of these properties to increase with an increase of λ and vanadium content has proved to be true only in the case of yield strength.

Properties of square profile with square hole [7]

The extrusion process of a square profile with a square hole was performed in two ways – without solution heat treatment on the press handling system (natural cooling) and with solution heat treatment on the press handling system using water wave.

The results relate only to the 6xxx alloy containing 0.2 wt% vanadium.

The photographs of macrostructure obtained in the square profiles with a square hole are shown in Figure 11. The coarse-crystal rim of less than 1mm can be seen on the profile extruded without water wave. Extrusion with cooling on the press handling system has made the grain size more uniform on the entire cross-section of the profile. In the case under discussion, grains in the central area were nearly two times smaller.

The size and distribution of the Mg_2Si phase precipitated during the homogenizing treatment of the examined alloy did not change regardless of the cooling technique applied after extrusion. Differences were found in the size of the strengthening phases, depending on the method of cooling after extrusion. During cooling and aging of the material solution heat treated on the press handling system, a decomposition of the solid solution took place, followed by the precipitation of fine-dispersed strengthening phases causing loss of alloy strength (Fig. 12). Mechanical properties are shown in Table 3. They confirm the declining tendency observed in alloy properties with the decreasing value of λ. The use of water wave slightly improved only the yield strength while reducing other material parameters.

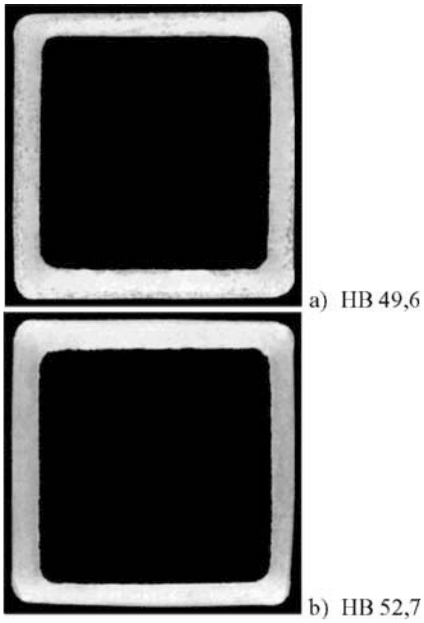


Fig.11 Macrostructure of square profiles with square holes extruded from the 6xxx alloy with an addition of 0,2% vanadium: a.) extrusion without water wave; b) extrusion with solution heat treatment on the press handling system using water wave – condition F

Table 3 Mechanical properties of a square profile with a square hole

Square profile with square hole						
Sample	**$R_{p0.2}$ [MPa]**	**R_m [MPa]**	**A [%]**	**$E_{Rp0.2}$ [J]**	**E_{Rm} [J]**	**E_{pek} [J]**
RV20	93	209	23,2	0,4	58	61
RV20 with water wave	97	198	19,8	0,4	50	50

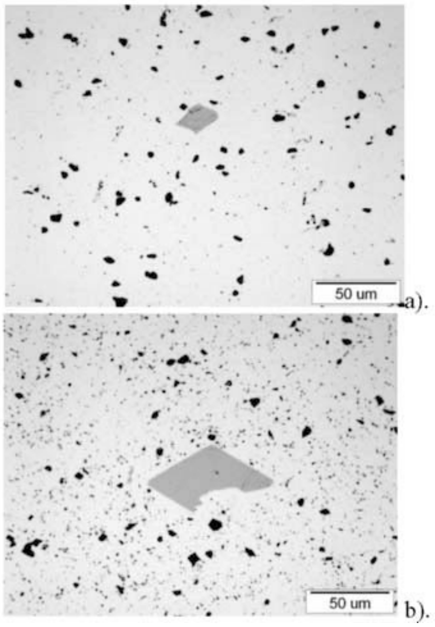


*Fig.12 Microstructure of square profiles with square hole extruded from the 6xxx alloy with an addition of 0.2% vanadium: a.) extrusion without water wave; b) extrusion with solution heat treatment on the press handling system using water wave – condition F (*x500).

Conclusions:

1. In the structure of ingots made from the 6xxx alloy with vanadium additions of 0.1-0.3 wt%, the following phases were identified: vanadium-containing iron phase AlFeSiV - $Al_{17}(Fe_{3,2}Mn_{0,8})Si_2$; ¸AlFe phase – Al_3Fe; $Al_{18}Mg_3V_2$ phase, and the strengthening Mg_2Si and AlFeSiV phases. The content of vanadium phases was proportional to the content of vanadium in alloy.
2. Vanadium addition was observed to refine the grains in ingots.
3. The homogenizing treatment of ingots had no effect on the morphology of large, block-shaped, vanadium precipitates.
4. Plastic working had no effect on the size and shape of the large precipitates of $V_2Mg_3Al_{18}$.
5. The properties of profiles extruded from the 6xxx series alloys with vanadium additions were characterized by higher values of elongation (with other mechanical properties kept at a similar level) compared to similar profiles made without the addition of vanadium.
   The use of water wave prevented the formation of a rim of coarse crystals on the profile cross-section.

References:

1. Ch.Leppin/ AluForm Congres 20-21 January 2004, Aluminium Crash Management and Simulation
2. Marzena Lech-Grega, Sonia Boczkal, Janusz Zelechowski „Phases with vanadium in industrial aluminum alloys" ECAA 2011 05-07.10.2011 Bremen, Germany
3. Marzena Lech-Grega, Zbigniew Zamkotowicz, Mariusz Bigaj, Maciej Gawlik "Wprowadzanie wanadu do kąpieli metalicznej oraz odlewanie wlewków z wanadem metodą półciągłą wraz z charakteryzacją ich struktury" paper submitted for publication in *Rudy i Metale*
4. M.Lech-Grega, W.Szymański, Z.Zamkotowicz, M.Biigaj, M.Gawlik "Struktura i własności wlewków ze stopów aluminium z dodatkiem wanadu do przeróbki plastycznej" 19th International Scientific and Technical Conference, KONTECH "Advanced Forming Technologies and Nanostructured Materials" Poznań-Opalenica 6-9.05.2012
5. Marzena Lech-Grega, Sonia Boczkal; "Iron phases in model Al-Mg-Si-Cu alloys"; *Materials Science Forum, Vol. 674 (2011), pp 135-140*
6. Marzena Lech-Grega, Wojciech Szymanski, Mariusz Bigaj, Maciej Gawlik, „Homogenisation of 6xxx alloy ingots with an addition of vanadium", paper accepted for 13th International Conference on Aluminum Alloys (ICAA13) 4-7.06.2013 Pittsburgh, USA
7. M.Lech-Grega, W.Szymański, B.Płonka, A.Kłyszewski, M.Biigaj, M.Gawlik "Wyciskanie i kucie stopów aluminium z dodatkiem wanadu" 19th International Scientific and Technical Conference, KONTECH "Advanced Forming Technologies and Nanostructured Materials" Poznań-Opalenica 6-9.05.2012
8. S. Camero, E. S. Puchi, G. Gonzalez, Effect of 0,1% vanadium addition on precipitation behavior and mechanical properties of Al-6063 commercial alloy, "Journal of Materials Science" 2006
9. J. L. Murray, AI-V (Aluminum-Vanadium), "JOURNAL OF PHASE EQUILIBRIA", Volume 10, Number 4, 351-357

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