

## MICROSTRUCTURE AND MECHANICAL PROPERTIES OF NANO-MICROCRYSTALLINE ALUMINUM ALLOYS

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**Abstract:** In past decades a new direction has developed in the production of aluminum products, applicable in automotive and aerospace industries. Composite alloys by adding inert nano- and microparticles are obtained. The influence of these particles on the structure of alloys at elevated temperatures is relatively poorly studied.

Composite extrudates from powders of low Si content are prepared. Addition of 2 wt. % TiC particles are used as a new phase in nano-microcrystalline aluminum alloys from the Al-Fe-V-Si system. We assume that the additive particles are inert and homogeneously distributed in the sample volume. Bulk samples have a high experimentally measured density of 2.9 g/cm<sup>3</sup> comparable to a calculated density value of 3.13 g/cm<sup>3</sup>. This indicates that the hot extrusion process provides good contact between the aluminum matrix and the TiC particles.

## МИКРОСТРУКТУРА И МЕХАНИЧНИ СВОЙСТВА НА НАНО-МИКРО КРИСТАЛНИ АЛУМИНИЕВИ СПЛАВИ

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**Ключови думи:** Нано-микрористални Al-Si сплави, рентгеноструктурен анализ, TEM, НаноСкан

**Резюме:** През последните години се развива ново направление в получаване на алуминиеви изделия, приложими в авто- и авиопрмишлеността. Получават се композиционни сплави, чрез добавяне на уякчаващи инертни нано- и микрочастици. Влиянието на тези частици върху структурата на сплавите при повишени температури е сравнително слабо изучено.

Изготвени са композиционни екструдати от прахове с ниско съдържание на Si. Добавка от 2 тегл. % TiC частици се използва като нова фаза в нано-микрористални алуминиеви сплави от системата Al-Fe-V-Si. Предполагаме, че частиците на добавката са инертни и хомогенно разпределени в обема на образците. Масивните образци са с висока експериментално измерена относителна плътност 2,9 g/cm<sup>3</sup>, съпоставима с изчислената стойност на плътността 3,13 g/cm<sup>3</sup>. Това показва, че процеса на гореща екструзия осигурява добър контакт между алуминиевата матрица и добавените частици TiC.

### 1. Introduction

Aluminum alloys with elements such as Si, Cu, Zn, Mg and others are used in industry because they combine low density with good mechanical properties [1, 2]. Their application in the automotive industry is steadily increasing. These alloys are used at operating temperatures of about 180 °C. Suitable alloying is used to increase their heat resistance. Typical examples are system alloys

(Al-Fe). They have a high heat resistance at Fe  $\geq$  7 to 10 wt. %. This determines their great application in the automotive and aviation industry.

In practice, as the most productive, fast melt melting, Planar Flow Casting (PFC) [3] and Gas Atomization [4] methods have been validated. As a result of the rapid crystallization, preforms with highly chopped structures, metal ribbons, slurries, flakes are obtained [5].

These blanks are compacted by plastic deformation processes at elevated temperatures such as hot extrusion. Heating structure is shown in [6].

This study analyzes the influence of TiC particles on the structure and physico-mechanical properties of Al-Fe-V-Si nano-microcrystalline alloys.

## 2. Experimental data

Nano-micro crystalline powders are compacted by hot extrusion. The powder is extruded at a temperature of 450 to 480 °C. By adding an inert additive (TiC) before the hot extrusion, composite extrudates are obtained. The composite extrudate has a size of  $\varnothing$  12 mm and a length of 1 m. From this, a composite tape is obtained through the PFC method [7]. The dimensions of the ribbon are 8 to 10 mm wide and 30 to 70  $\mu$ m thick. A small sample for study with Nanoscan and the test strip is given in Fig. 1.

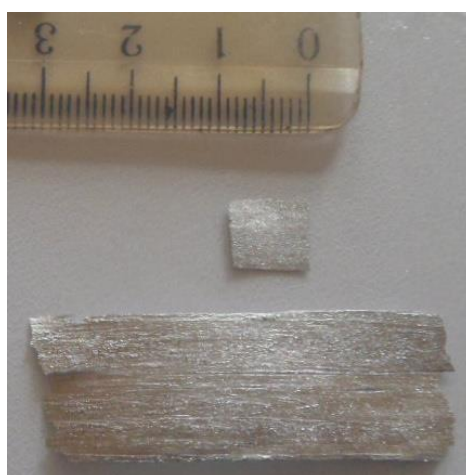


Fig. 1. Micro-nanocrystalline ribbons from the system AL-Fe-V-Si

The ribbons are characterized by two different surfaces. On the side of the cooling disk the surface is smooth (WS). The other side is visibly rugged (AS). Samples of the starting microcrystalline powder Al-Fe-V-Si were obtained:

Sample 1 – ribbon AL-Fe-V-Si

Sample 2 – ribbon AL-Fe-V-Si with TiC particles

Table 1. Chemical composition of the samples' surface in wt. %

wt %	Al	Fe	V	Si	Ti
Sample 1	86.52	8.60	1.55	3.13	-
Sample 2	88.83	8.86	1.53	-	0.78

From previous data [1, 5], the phase composition of the surface of the ribbons is determined (Table 1). The absorbed amount of TiC in the strip is about 2 wt. % with Ti  $\sim$  1% concentration.

In heat treatment, a staged decomposition, mainly in the nanosecond, of the saturated Al solid solution which is associated with the final microstructure formation (Table 2) occurs. In the first stage, silicon particles are separated, and the grain parameter of the pressurized solid solution gradually increases and reaches equilibrium values.

A two-phase microstructure is obtained. When additional alloying elements are present, the system becomes three-phase, separating a new intermetallic phase.

Table 1. Scheme of microstructure formation in nano-microcrystalline Al-Si alloys with addition of an alloying element Sb

I stage	II stage	III stage	IV stage
Pressed Al-solid solution	Si phase	IM iron containing phase	AlSb phase
			T > 540 °C

The presence of additional alloying elements complicates the microstructure shaping mechanism by adding an additional step leading to the growth of a second intermetallic phase. The separated secondary Si during the first decomposition step of the Al solid solution serves as a support for the growth of the corresponding intermetallic phase.

Complicating the composition of the starting alloys is an additional source for producing nano-micro alloys with unique properties [8].

### 2.1. OM and TEM analyses of samples

Extruded composite specimens have a nano-micrograin microstructure with a uniform phase distribution throughout the volume (Fig. 2).

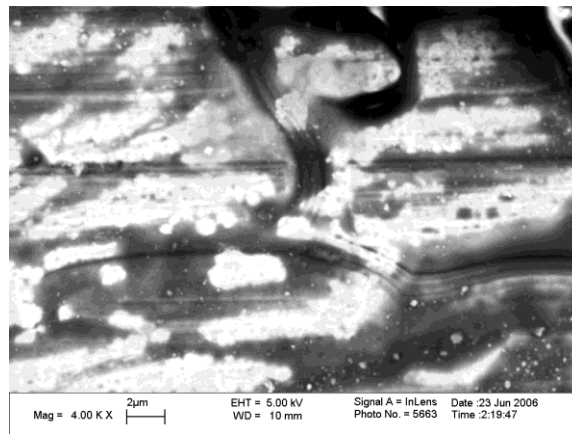


Fig. 2. Metallographic structure of ribbon

The figure shows a homogeneity in the distribution of the TiC particles in the sample volume 2 (the light points in the figure) along the whole section of the ribbon.

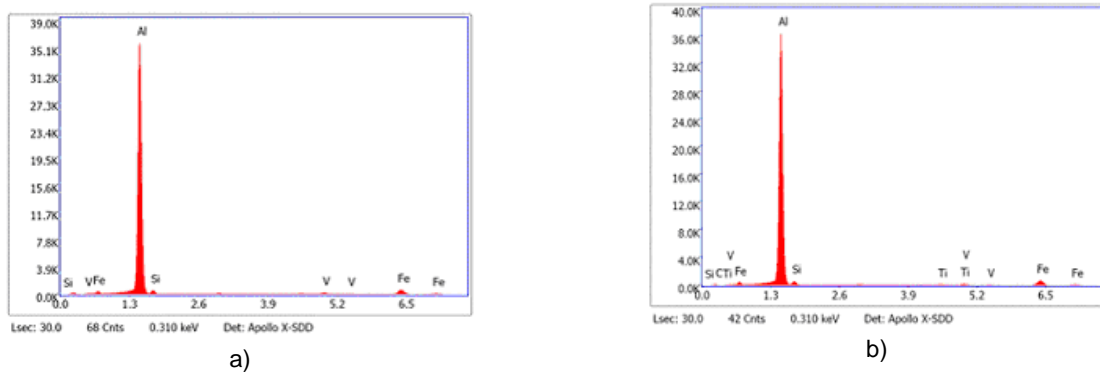


Fig. 3. EDS spectrums of: a) Sample 1; b) Sample 2

The TiC particles are spherical in shape. Composite samples 1 and 2 are tested by TEM with EDX to specify the structure and composition, Fig. 3 a) and b), Table 2 a) and b).

The smooth surface of the ribbons is monitored by a nanoparticulate solid aluminum solution with a grain size of 50–70 nm and intermetallic particles up to about 10 nm.

Table 2. Chemical composition of: a) Sample 1; b) Sample 2

Element	Weight %	Atomic %	Element	Weight %	Atomic %
AlK	88.08	91.76	C K	2.57	6.00
SiK	1.89	1.93	AlK	81.89	85.07
V K	1.87	1.05	SiK	1.92	1.92
FeK	10.19	5.25	TiK	0.96	0.56
			V K	1.91	1.05
			FeK	10.75	5.40

a)

b)

The grid parameter of the oversaturated solution is smaller than the equilibrium value.

## 2.2. SPM analysis

NanoScan, FSBI TISNCM, Russia is a microscope that allows the obtention of three-dimensional surface topography images in the in tapping mode with a diamond tip (Fig. 5). The scanning is performed attached to a piezoceramic probe [9]. During the scanning process, the frequency is kept constant. This mode allows studying the structure of multiphase materials, as well as the mechanical heterogeneity distribution on the surface.

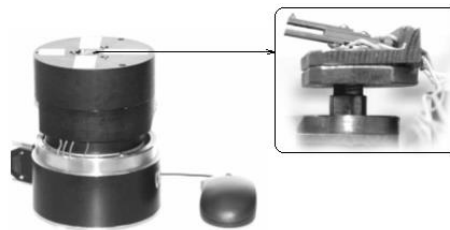


Fig. 5. Microscope Nanoscan with a piezoceramic probe

With additional NanoScan research, the effect of the composite additive on the properties of the two ribbons is demonstrated. Figure 6 shows the boundary of the sample 2.

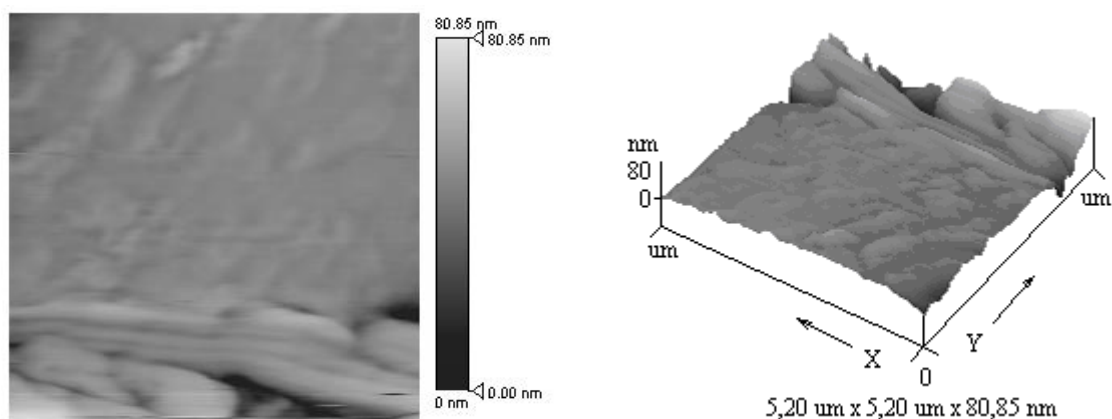


Fig. 6. Surface topography of a ribbon 2, obtained by scanning probe microscope NanoScan: a) 3D and b) 2D images

Surface roughness was characterized by calculating the roughness parameters: Ra, Rms, Rz and maximum elevation Max h. Average arithmetic roughness of the surface Ra - average roughness describes different shape, dimensions and direction of furrows, recesses and projections that form the

relief of a given surface. Mathematically represents the sum divided by their number. Higher value means a coarse surface. In mathematics, Rms - root mean square roughness is a statistical measure of the magnitude of the surface roughness variable. It is always greater in value than Ra, being more sensitive to more extreme points. Ten point average roughness Rz is the difference in height between the average of five highest peaks and five lowest valleys in the surface profile. Rz is more sensitive to occasional high peaks or deep valleys on surface profile analysis of samples

Typical for the sample is the magnitude of the Max h. The data in Table 3 are values averaged over 10 measurements.

The use of the presented methods complements the information on the structure and the surface layer of the samples.

Table 3. Roughness statistics for samples

Sample	Ra [nm]	Rms [nm]	Rz [nm]	Max h (Z) [nm]
1	8.02	10.08	27.22	68.38
2	8.11	13.01	38.87	81.23

The Z difference between the sample surfaces is 12.85 nm. The roughness Ra is greater (0.09 nm) for sample 2 with TiC (Fug. 8, Table 6).

Increasing roughness in the presence of inert particles influences by creating additional stresses in the material.

### 2.3. Microhardness

The structure of nano-microcrystalline bands determines their mechanical properties. Several nano-microprint bands of different composition were studied. The micro-stability of the test ribbons varies depending on their chemical composition. The highest values of micro-hardness were measured in Fe-doped ribbons.

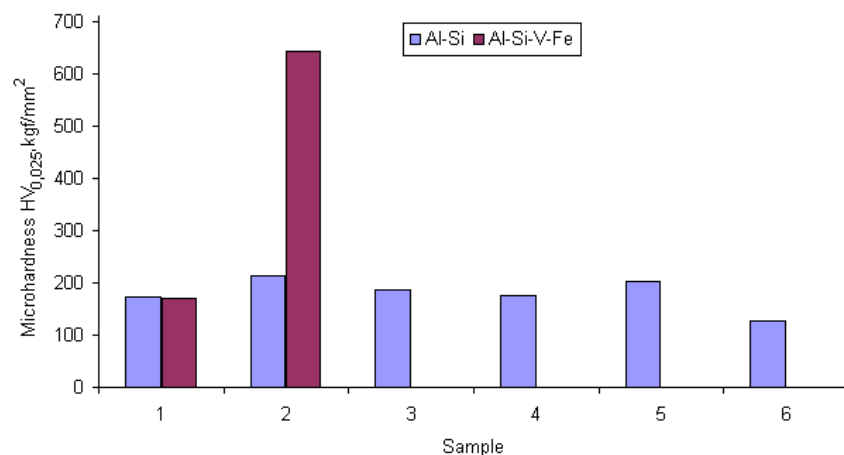


Fig. 6. Microhardness of test ribbons from the system Al-Si and Al-Si-V-Fe

We investigated a series of rebbons: Al alloy double alloy alloys, samples 1 to 6 and high alloy alloys with 8–9 % iron samples 1 and 2. Micro-hardness does not change much. In all, iron phases are observed. Highest iron have experimental samples from the quartz Al-Fe-V-Si alloy, but due to the peculiar way of production, there are no needle-like phases and high micro-stiffness values.

Hardness measurements of the resulting composite bands were performed.

Micro-hardness of alloys with twice the silicon content is twice as high, which confirms the idea that the emergence of an additional phase leads to improved mechanical performance.

The properties of the alloys are verified by thermal treatment. This process influences the degree of solidification in the phase-forming processes.

## Conclusions

Al-Fe-V-Si metal matrix composite materials are presented. Consideration is given to the possibility of inserting fillers (nano and micro particles) for further dispersion of the alloy.

1. The high content of Fe in conventional alloys results in the formation of large, needle-like iron phases. The structure of the cast articles is fragile. The shape and dimensions of the phases formed depend on both the chemical composition and the rate of crystallization. Additives from microcomponents as well as suitable casting and heat treatment technologies lead to the elimination of intermetallic phases and a significant improvement in the mechanical properties of the parts. This technology allows the processing of secondary alloys with high iron content and other alloying elements, which helps to protect the environment.

2. Rapid hardening methods result in new alloys with increased mechanical performance. This allows the introduction of advanced technologies for the processing of secondary aluminum alloys, and the harmful impurities are transformed into necessary enhancing and / or alloying phases.

In one case, microcrystalline bands are obtained, and in the other - crystalline powders with micron particle sizes, with substantial ejection of the phases in them. Aluminum alloys thus obtained can be found in practical applications in machine-building after compacting to massive blanks. For this purpose, the methods of plastic deformation and in particular hot extrusion are used.

3. Because of the high melt cooling rates, these materials are far from equilibrium. Their structure is composed of a saturated aluminum matrix and reinforcing, intermetallic phases with sizes from several nm to several  $\mu\text{m}$  (depending on the chemical composition of the alloys). Alloy etching is based on a relatively high amount of fine particles that are stable at elevated temperatures due to the low diffusion rate and limited solubility of the alloying elements.

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