X-ray diffraction studies of rapid cooled Al-V and Al-Fe-V alloys

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ABSTRACT

Purpose: of this paper is to deep and more complete knowledge about the features of phase and structure formation in Al-based alloys with transition metals (TM) Fe and V at rapid cooling from melt. It is known, that nonequilibrium synthesis conditions of such alloys lead to quasicrystalline, amorphous or metastable phases formation, which can significantly improve the physical-chemical properties and first of all the mechanical ones. But understanding of compositional dependences of structure features at formation under nonequilibrium conditions and the correlation of these dependences with physical properties of alloys is far to be clear.

Design/methodology/approach: Structure of Al-enriched Al-V, Al-V-Fe rapid cooled alloys was studied by X-ray diffraction method. In order to estimate the influence of structural state of alloy on the mechanical properties the integral microhardness was studied by Vickers method.

Findings: Two quasicrystalline icosaedral phases with different cell parameters are revealed in ternary alloys $Al_{100-3x}V_{2x}Fe_x$ (x=2-4). Increasing of transition metal content promotes the formation of phase with higher quasicell parameter embedded in amorphous matrix. With increasing of the transition elements total content from 6 up to 12 at. % the microhardness of alloys increased gradually from 867 to 3050 MPa.

Research limitations/implications: Research of nonequilibrium alloys revealed crystalline structure of Al-V alloys and quasicrystalline embedded in amorphous matrix of Al-Fe-V ternary alloys. Obtained results suppose that further structure and physical properties studies of Al-Fe-V alloys will allows to find the conditions to control the producing of materials with desired properties.

Practical implications: Using of rapid cooling method for synthesis of Al-enriched Al-Fe-V alloys give an opportunity to produce alloys with significantly improved mechanical properties.

Originality/value: Nonequilibrium conditions of cooling allow significantly changes the structure and properties.

Keywords: Al-based alloys, Quasicrystals, Rapid cooling, Microhardness

Reference to this paper should be given in the following way:

O. Shved, S. Mudry, V. Girzhon, O. Smolyakov, X-ray diffraction studies of rapid cooled Al-V and Al-Fe-V alloys, Journal of Achievements in Materials and Manufacturing Engineering 96/1 (2019) 5-11.

MATERIALS

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1. Introduction

Ternary Al-Fe-V system attracts the attention of researchers especially its aluminum enriched alloys because they are interesting for technology application as light materials with high strength, good ductility or toughness [1,2]. It is shown by Inoue et al. [3] that further significant improvement in strength of rapidly solidified Al-Fe-V alloys may be reached in alloys containing nano-quasicrystalline phase. It was reported by Kim et al. [4] that forming a precipitation of nanometer-sized *fcc*-Al based solid solutions in the amorphous matrix leads to increasing of hardness. The solubilities of Fe and V in Al are very limited when the alloy is produced by means of the conventional method with low cooling rate, e.g. Murray [5]. In order to increase the solubility of Fe and V in aluminum the rapid cooling from the liquid state is commonly used that allows attaining the solubility of V up to 1.25% and Fe to 4.4% [6]. The solubility of iron in aluminum is very limited and it is increasing upon rapid cooling up to 15 at.%. The lattice parameter of Al decreases down to 4.012 A upon rapid cooling. Although these metals have the similar crystalline structures (*ccc* in Fe and V; bcc in Al) and the difference of atomic radii (r_{Al} = 0.125 nm, r_{Fe} = 0.140 nm, r_V = 0.135 nm) is not so large as well as electronegativity differences (χ_{Al} = 1.61, φ_{Fe} = 1.83, χ_V = 1.63) the solubilities in solid state are such low even at elevated temperatures and their maximum values are about 0.3 at.% at 930 K. Maybe some difference between *ccc*- and *bcc*- structures is one of the reasons that Al-Fe-V alloys maintain high strength up to 573 K due to the presence of thermally stable second phase with nanoscale size. It was established by Sokolovskaya et al. [7] that the Al_3Fe and $Al_{10}V$ are in equilibrium with an aluminum solid solution. These two compounds are also components of eutectic formed at 610 K and 83 at. % of Al.

It is known that high cooling rate of melt may possible to obtain the quasicrystals (QC), particularly the icosahedra phases. The different methods are used for this purpose; first of all, it is melt spinning, mechanical activation, deposition [8,9] and laser doping propozed by Girzhon et al. [10].

Al-TM binary systems (TM = V, Cr, Mn, Fe, Co, Ni) currently are the most studied in order to understand the mechanism of QC formation process. In particular, Chen et al. [11] showed that ability to the formation of binary QC-phases decreases with increasing of the 3d-electrons number in TM. Lawther et al. [12] showed that cell parameters for listed above set of elements also decrease gradually. Unfortunately to update no correct interpretation of phase formation processes, at which

QC-phases are created, especially at crystallization conditions, far from equilibrium ones.

At present time it is known that at a high cooling rate of Al-V-Fe melts it is possible to obtain the icosahedra phases the features of formation of which are studied by many authors [13-17] to give the deep and complete knowledge. For that reason the aim of this work was to study the structure formation features of QC-phases in $Al_{100-3x}V_{2x}Fe_x$ alloys, cooled from liquid state at the same conditions.

2. Experimental

Two binary – $Al_{91}V_9$, $Al_{90}V_{10}$ – and four ternary alloys – $Al_{94}Fe_4V_2$, $Al_{94}Fe_2V_4$, $Al_{91}Fe_3V_6$, and $Al_{88}Fe_4V_8$ – as ribbons have been obtained by the method of standard planar flow casting in the controlled atmosphere of inert gas with a rapid cooling rate of 10^6 K/s. The casting chamber was preliminary out gassed and then filled with argon up to 103 Pa. The melt was inductively overheated and then injected onto rotating copper wheel.

X-ray diffraction method (XRD) was used for structure investigation in the result of which by analysis of diffraction patterns the main structural parameters were determined (Cu $K\alpha_1$–radiation, λ=1.540559 Å). Phase analysis was performed by least squares refinements of XRD data using PowderCell program. Structural parameters were determined by the line broadening analysis with the program FullProf.2k [18].

Microhardness was measured by Vickers microhardness tester PMT–3 with pyramidal diamond indenter using the applied force of 15 g. The value of microhardness was calculated by the formula:

$$H_V = \frac{1.8544P}{D^2},\qquad(1)$$

where D is the average length of the diagonal left by the indenter, mm^2, P – the force applied to the diamond, H.

3. Results

Phase content of rapid cooled $Al_{90}V_{10}$ alloy deviated from the composition, which should be according to the equilibrium phase diagram given by Okamoto [19]. As follows from XRD analysis it consists of Al, Al_3V (Al_3Ti structure type) and $Al_{21}V_2$ ($Al_{21}V_2$ structure type) phases (Fig. 1). According to [19] alloy of such concentration at room temperature should consist of $Al_{21}V_2$ and $Al_{45}V_7$ phases.
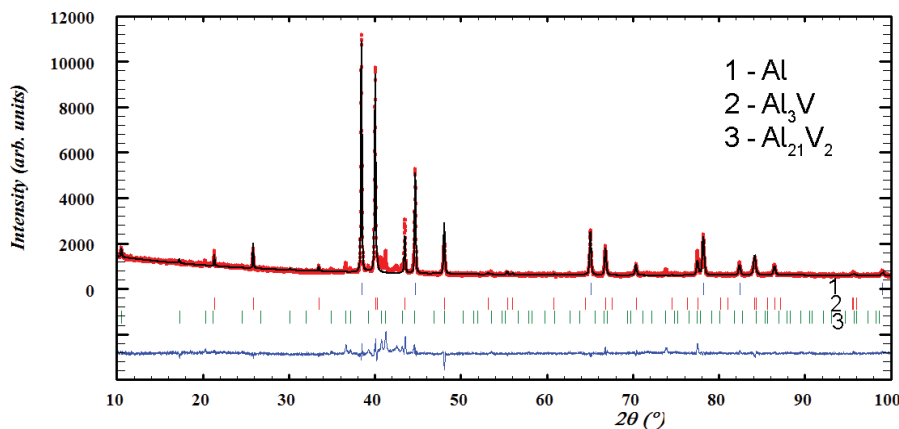
Fig. 1. X-ray powder diffraction intensity data of rapidly cooled $Al_{90}V_{10}$ (*a*). The vertical bars indicate the calculated Brag positions of peaks of phase components of alloys; the dots shown experimental data; solid lines – calculated, and $Y_{exp.}$-Y_{calc} is the intensity difference between intensity curves

Cell parameter, calculated from values of peak positions for Al(V) solution equals to 0.40599 nm and is very close to one of pure aluminum (0.40488 nm). Some increase of lattice parameter for Al(V) solution comparing to aluminum is clear because the atomic radius of vanadium is larger than one of aluminum. Assuming that atoms of vanadium are diluted in the Al matrix then peaks, related with intermetallics should be of a very low intensity that is not observed in experimental diffraction patterns. Contrary, of high intensity diffraction peaks, corresponding to AlV_3 intermetallics are pronounced. Such features of diffraction pattern can be explained if take into account the structural characteristics in liquid state and structure change at cooling from the melt to solid phase. As follows from diffraction studies and physical properties measurements most of the metallic liquid alloys have a cluster structure [20]. Such kind structure is supposed to be also in Al-V alloys. Since the rapid cooling process is related with some retaining of liquid structure at transition to solid one it is possible to suppose that in liquid state an atomic arrangement of Al(V) alloy is an atomic solution, but due to the tendency to interaction of unlike kind atoms in binary Al–V system, increasing with cooling, the atomic arrangement will be somewhat another than in atomic solution, especially at temperatures near solidification point. Taking into account the results of XRD studies we supposed that in of such kind systems exist two kind clusters: Al(V) clusters, forming the matrix and chemically ordered clusters Al_nV embedded into this matrix. Upon rapid cooling these clusters increase their size, forming a mixture of crystals of nanoscale size, values of which have been determined from the half width of diffraction peaks and were found to be 45 nm.

At studying of ternary alloys the content of Fe to the content of V ratio is commonly chosen as 1:2 and only in one case ($Al_{94}V_2Fe_4$) this relation was opposite.

XRD analysis has shown that diffraction pattern of $Al_{94}V_4Fe_2$ alloy, which consists lower total amount of transition metals, is significantly different than previous one (Fig. 2a) and besides diffraction peaks of Al the high-intensity peaks from icosahedra i_1–phase with quasi-cell parameter a_q = 1.785 nm are pronounced. The identification of reflexes for i_1–phase was carried out with using (N, M) numbers, which were suggested by Cahn et al. [21] where interplanar distances have been determined from equation

$$d / n = a_q \big/ \sqrt{N + M\tau} , \qquad (2)$$

where $\tau = \left(\sqrt{5}+1\right)/2$ – gold ratio.

It should be noted that there is an asymmetry of maxima (18, 29), (20, 32) and (52, 84), with a shoulder on higher angles side, that may be evidence about the existence of once more icosahedra i_2–phase. The variation of vanadium to the iron ratio ($Al_{94}V_2Fe_4$) did not lead to a qualitative change of diffraction pattern (Fig. 2b). However, the diffraction peaks from QC-phase were notably shifted to larger values of diffraction angles and cell parameter was found to be 1.755 nm. A significant difference of quasi-cell parameters for $Al_{94}V_4Fe_2$ and $Al_{94}V_2Fe_4$ alloys allowed us to state that in Al-V-Fe system it is possible the formation of various icosahedra QC-phases (i_1 and i_2). Thus, mentioned above asymmetry of peaks for i_1-phase of the $Al_{94}V_4Fe_2$ alloy can be related with the coexistence of two QC-phases in this alloy with different quasi-cell parameters values.

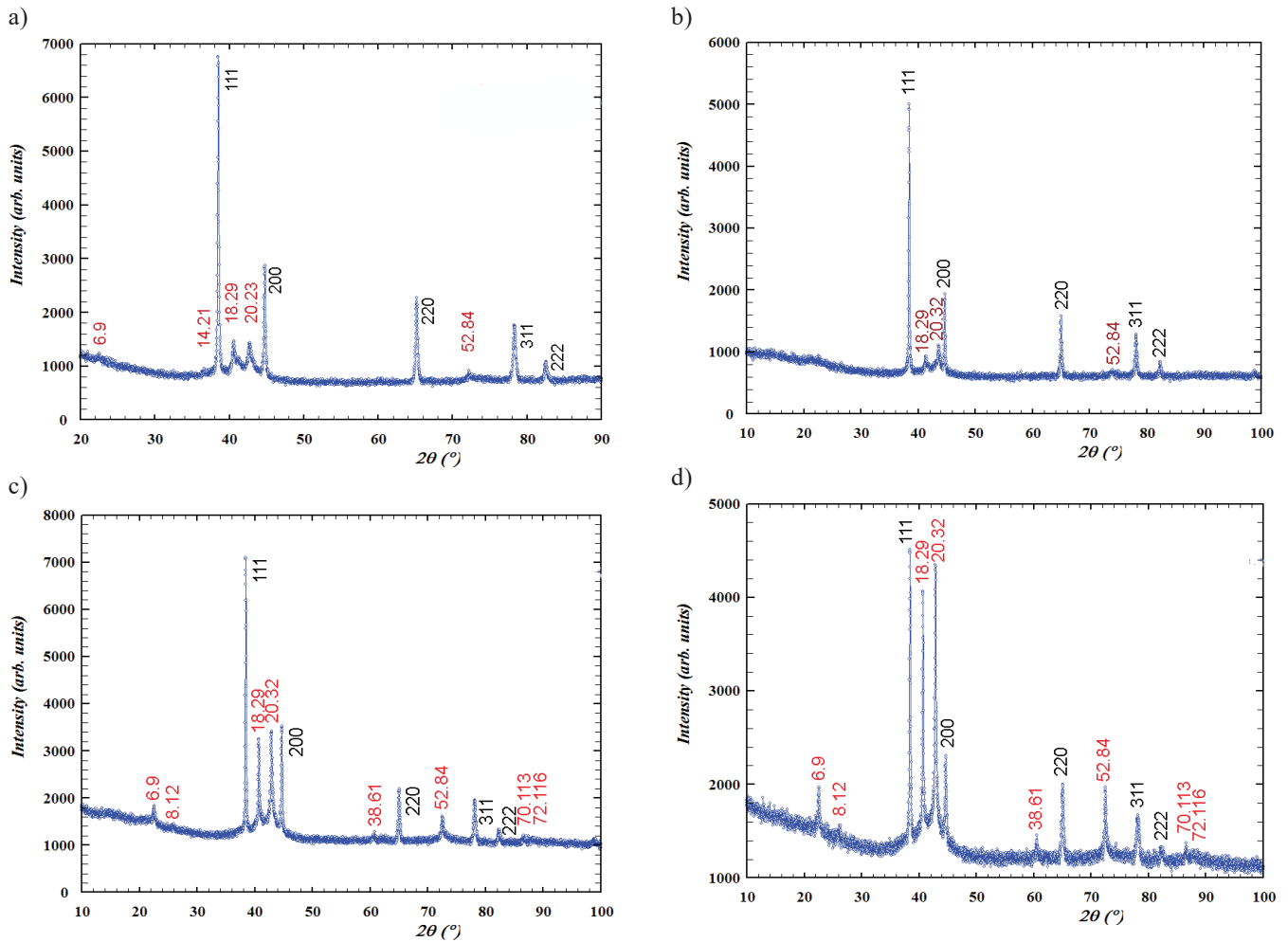Fig. 2. X-ray powder diffraction intensity data of rapid cooled $Al_{94}V_4Fe_2$ (a), $Al_{94}V_2Fe_4$ (b), $Al_{91}V_6Fe_3$ (c) and $Al_{88}V_8Fe_4$ (d) samples. The black indexes indicate *fcc*-Al reflexes, the red – peaks of i_1–phase and dark red – peaks of i_2–phase

The increase of a total number of transition metals ($Al_{91}V_6Fe_3$) caused the increasing the number of i_1–phases with quasi-cell parameter $a_q = 1.785$ nm (Fig. 2c). Notably, that diffraction pattern also revealed an amorphous halo of low intensity. The possibility of some fraction of amorphous phase formation was noted early by [15, 17].

Alloy, containing the highest content of transition metal ($Al_{88}V_8Fe_4$) consisted of three phases: Al, i_1–phase with the same parameter $a_q = 1.785$ nm and amorphous phase (Fig. 2d).

Since the content of Al prevails in alloy most attention was paid to change of crystalline grains sizes for its solid solutions as dominant phase of such alloy. In binary alloy, this parameter was found to be about 45 nm. This value is significantly less than in conventional Al-based alloys. It is interesting to study how phase content and structure

parameters change if some part of the iron atoms is replaced by ones of V. In this case, the diffraction pattern shows the significant change. Cell parameter of Al in $Al_{94}V_4Fe_2$ alloy is slightly less comparing with others. Most probably Fe-atoms are diluted in Al and as result, we observe also further decrease of nanocrystals size (Tab. 1). In this case, the lattice parameter of Al is almost the same, but the size of Al-based crystallites is more reduced and equals to 31.7 nm. Therefore in Al corner of the phase diagram, the sensitivity of crystallite size to the phase content of the alloy is very high and allowed to obtain the crystalline alloys with the low size of crystalline grains. Besides the size of crystallites, it was possible to calculate also values of microstresses (Tab. 1). It can be seen that this parameter shows the minimum value for the alloy with the highest crystalline grain size. Certainly, these two

parameters are related to the mechanical properties of the alloy. In order to find the dependence between the size of crystallites and mechanical properties, we have measured also microhardness. It can be seen (Tab. 1) that values of microhardness, obtained by (1) increase with reducing of crystallites size that is typical for metallic alloys.

Table 1.
Phase characteristics and Vickers microhardness of Al–V and Al–Fe–V as rapid cooled alloys

Alloy	Phase content	Lattice parameters, nm		$L,^{1}$	$E,$	$H_V,$
		a	c	nm	$\times 10^{-4}$	MPa
$Al_{90}V_{10}$	Al	0.40505(1)	-			
	Al_3V	0.37814(1)	0.83145(4)	45.1	1.9	755
	$Al_{21}V_2$	…	….			
$Al_{94}Fe_4V_2$	Al	0.40521(5)	-			
	i_2	1.785	-	35.6	2.6	867
	amorph. ph.					
$Al_{94}Fe_2V_4$	Al	0.4045(1)	-			
	i_1	1.785	-	31.7	3.1	1540
	i_2	1.755	-			
$Al_{91}Fe_3V_6$	Al	0.4052(1)	-			
	i_1	1.785	-	30.6	2.7	2810
	amorph. ph.					
$Al_{88}Fe_4V_8$	Al	0.4052(2)	-			
	i_1	1.785	-	27.3	4.1	3050
	amorph. ph.					

[1] Grain size and microstresses were determined from the width of the (111) and (222) reflexes of Al phase

4. Discussion

In order to understand the structural features in alloys under investigation, we should interpret the experimental data. According to Okamoto [19], $Al_{90}V_{10}$ alloy at room temperature should consist of $Al_{21}V_2$ and $Al_{45}V_7$ phases. The discrepancy between phase content, obtained from experimental data and one predicted by equilibrium phase diagram is supposed to be caused that at equilibrium crystallization the solid state formation during cooling occurs due to few peritectic reactions. In our case due to rapid cooling the high temperature Al_3V phase had no time to be decomposed and as result, the melt was Al-enriched promoting the crystalline aluminum formation. It should be noted that the formation of QC-phase was not fixed. It could indicate that the chosen cooling rate of melt was not high enough to form this phase.

A detailed description of general features of QC-formation processes in binary alloys of Al-TM systems have been considered in [11] by Chen et al. Authors of this work had proposed the optimal ways to choose the stoichiometry of alloys with the ability to QC formation. Such choosing was based on the fact that cells of some crystalline phases consist of groups of atoms with icosahedra structure. In the interior of icosahedra is an atom of a transition metal. Besides, one atom of TM is located in one of the icosahedra tops in order to have an atomic distribution with most dense packing. Such atomic groups are marked as $[TM_2Al_{11}]$. QC-phases are created by means of gluing of such groups with help Al-atoms forming $([TM_2Al_{11}]Al_1)$, or with help of TM-atoms that results in the formation of $([TM_2Al_{11}]TM_1)$. In Al–V system such phases, which consist icosahedra groups are $Al_{21}V_2$, $Al_{23}V_4$, $Al_{45}V_7$, whereas in Al-Fe system such phase is $Al_{13}Fe_4$. At least in Al-V-Fe ternary system, there are such QC-phases:

1) $[V_2Al_{11}]Fe_1$, which has the stoichiometry, close to the stoichiometry of $Al_{80}V_{12.5}Fe_{7.5}$, obtained as single-phase and containing only icosahedra QC-phase Lawther et al. [12];
2) $[Fe_2Al_{11}]V_1$, in which the ratio of iron to vanadium atoms is reciprocal value.

Therefore it is simply to explain the presence of two icosahedra QC-phases in the alloy of A-V-Fe system that was established by experiment. These phases have different quasi-cell parameters and depending on a ratio of vanadium to iron atoms, the formation of QC-phase from $[V_2Al_{11}]Fe_1$ or $[Fe_2Al_{11}]V_1$ atomic groups occurs.

The question arises what is the reason of simultaneous existence of two QC-phases in the $Al_{94}V_4Fe_2$ alloy. Due to high Al content, the mean interatomic distance between TM-atoms is sufficiently large. This is just a reason that probability to find the atoms of V or Fe in centers of icosahedra groups and gluing process can occur also with help of vanadium or iron atoms. Theoretically in such solution can be the next cases:

1. $[Fe_2Al_{11}]Fe_1$;
2. $[Fe_2Al_{11}]V_1$;
3. $[Fe_1V_1Al_{11}]Fe_1$;
4. $[Fe_1V_1Al_{11}]V_1$;
5. $[V_2Al_{11}]Fe_1$;
6. $[V_2Al_{11}]V_1$.

Implementation of the binary stoichiometry of types 1 and 6 in diluted solution is of low probability. Thus there are two possible variants of stoichiometry – $Al_{11}V_1Fe_2$ and $Al_{11}V_2Fe_1$, which correspond to types 2 and 5. In such a case close to them corresponding QC-phases (i_1 та i_2) can compete with each other at crystallization.

It is of interest to analyze the behavior of quasi-cell parameter. Lawther et al. [12] shown, that the dependence of quasi-cell parameter on number of Fe to V atoms ratio in $Al_{86}V_{14-x}Fe_x$ alloy has been studied and for this parameter was used method described by Elser [22]. Taking the results of experimental studies and using this method for determination of a quasi-cell parameter of Cahn et al. [19] it is possible to estimate the number of Al to V ratio in QC-phases – i_1 and i_2. For i_1–phase, this ratio leads to the stoichiometry of $Al_{86}V_{9.3}Fe_{4.7}$, and for i_2 one to the stoichiometry of $Al_{86}V_{4.3}Fe_{9.7}$. In other words, such ratio is 2:1 for i_1 phase and 1:2 for the i_2–phase.

The fact that slight change of the number of V to Fe ratio can promote the qualitatively different phase content is confirmed by Inoue et al. [15], whereby authors are shown that rapid cooled $Al_{95}V_3Fe_2$ alloy consisted of two phases: Al and i–phase. Maxima positions for i–phase confirmed that its quasi-period was close to 1.755 nm.

A slight increase of V atoms content ($Al_{94}V_4Fe_2$), at the same conditions of its manufacturing, caused the change of phase content, particularly the appearance of reflexes of Al and amorphous halo have been observed by [15]. On other hands, the authors established that $Al_{93}V_5Fe_2$ alloy structure was again two-phase – Al and i–phase, whereas the quasi-cell parameter for QC-phase was close to 1.785 nm. The formation of amorphous phase at some definite chemical composition can be caused by competition of two QC-phases with different quasi-cell parameters. It should be noted that total increase of TM in the alloy (at constant ratio the number of iron to vanadium atoms as 1:2) in fact did not affect the quasi-cell parameter of i_1-phase,

confirming an assumption about simultaneous coexistence both quasicrystalline phases in the $Al_{94}V_4Fe_2$ alloy.

Total microhardness of alloys notably depended on their chemical composition. Namely by increasing the total content of transition metals microhardness was increased gradually (Tab. 1).

Such a feature has a simple interpretation and is related with increasing the fraction of quasicrystalline and amorphous phases when the content of transition metals increases. Just the fraction of amorphous phase is most responsible for the microhardness increasing.

5. Conclusions

1. Rapid cooling from liquid state of $Al_{94}V_4Fe_2$ alloy with a low content of transition metals reveals the simultaneous formation of both i_1 and i_2 QC phases with different quasi-cell parameters.
2. Increasing TM content in $Al_{100-3x}V_{2x}Fe_x$ alloys promotes the formation of i_1 quasicrystalline phase with much higher quasi-cell parameter and amorphous phase formation that may be the result of structure competition at solidification of the alloy.
3. The microhardness of Al-enriched Al-Fe-V rapid cooled alloys increases with increasing of total TM content in the alloy.

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