

Formation of Phase Icosahedral and Decagonal Quasicrystalline Alloys $\text{Al}_{62,2}\text{Cu}_{25,3}\text{Fe}_{12,5}/\text{Al}_{65}\text{Ni}_{15}\text{Co}_{20}$ Influence on The Oxidation

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Abstract

In this article we will cover a study of the formation of icosahedral and decagonal phases two quasicrystals $\text{Al}_{62,2}\text{Cu}_{25,3}\text{Fe}_{12,5}$ And $\text{Al}_{65}\text{Ni}_{15}\text{Co}_{20}$, and the influence of oxidation in this alloy. For this purpose research used the diffraction of X-ray (XRD), scanning electron microscopy (SEM)/Energy Dispersive Spectroscopy (EDS), Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TG). The results displayed found aspects of morphological structural as well as the surface of the two compositions of quasicrystals, these were prepared and obtained in electric arc furnaces and induction and arc. Oxidation of $\text{Al}_{62,2}\text{Cu}_{25,3}\text{Fe}_{12,5}$ alloy, intermetallic phases presented with combinations of alloying elements and above 675°C it was observed that the crystalline phase is stable. In icosahedral phase oxidation of aluminum forms a dense layer on the passivating outermost surface of the quasicrystal which causes depletion in both copper and iron. In $\text{Al}_{65}\text{Ni}_{15}\text{Co}_{20}$ nominal composition of oxygen interaction occurs on the surface of symmetry 10 times plane perpendicular vector. The formation of a thin film of aluminum oxide having well-ordered hexagonal structure and with the opposition area decagonal phase with the lateral size of approximately 35 Å.

Keywords : quasicrystals; phases; icosahedral; decagonal; aluminum oxide

Introduction

The quasicrystals are interesting materials with scientific research more than two decades. However, arouse the curiosity of researchers in search of new knowledge on the subject. But the advantages are related to the quasicrystalline alloy provides in terms of applications in the technological field. Discovered by (Shechtman 1984), the quasicrystals is solid with long-range crystallographic prohibited rotational symmetries and aperiodic in the atomic arrangement, with unusual physical properties.⁽¹⁾

The compositions of the quasicrystalline alloys are usually ternary or binary; some compositions have been research and success for certain uses. Among the other compositions of quasicrystals, of course the most investigated to know; AlCuFe, AlMnPd, AlNiCo, Al-Pd-Mg, Al-Cu-Co, Al-Co-Fe-Cr. These quasicrystalline alloys have shown good properties

such as high hardness, low electrical and thermal conductivity, low surface energy, accompanied by a low coefficient friction, strong corrosion resistance and good hydrogen storers for use in catalysis.⁽²⁾

Quasicrystals are thermodynamically stable under conditions of high temperatures above 800°C under an inert gas atmosphere. But the system with AlCuFe elements shows the icosahedral phase, one of the most researched compositions by scholars on this subject. However, for the formation of quasicrystals there are other important phases in quasicrystalline alloy process. Typically, intermetallic, amorphous and crystalline phases are systems based on combinations with other elements and Al, Co, Ni, Cr, Fe, Mg, Zr, Ti, Zn e Cu, are formed due to phase transitions existing between one phase and another on a solid state reaction.

The quasicrystalline alloys exhibit rotational and structural symmetries with five, eight, ten and twelve have formations icosahedral, decagonal

and dodecahedron in general dependence of a unit cell structure.⁽³⁾

The solid crystalline which are usually studied in research centers is; $Al_{65}Cu_{22.5}Fe_{12.5}$, and interestingly the quasicrystalline phase is the result of a peritectic reaction phase β_2 - $AlFe_3$ and the remaining liquid. But is $AlCuFe$ formation is rich in aluminum and of course the coexistence of icosahedral quasicrystalline phase ψ with other phases belonging.

The mechanical conventional methods of obtaining quasicrystals are influenced by synthesis. Therefore, allows the preparation of widely varying composition and existence of phases, making it easy to technology.

The quasicrystals have two structural namely; Icosahedral ($AlCuFe$) and decagonal ($Al-Ni-Co$, $Al-Cu-Co$) in this training phase related particulate grain and the preparation process. As well, the difference in concentrations of the two quasicrystalline materials to be balanced by interdiffusion in a transitional region, thereby forming to-one quaternary alloy $AlCo$ (Cu, Ni) of the crystal in a narrow region.

The alloy composition $AlCuFe$ whose main phase is icosahedral, arising from other previous phases. In this mechanism the cubic phase β - Al (Fe, Cu), predominates is the main driving force in the formation of the icosahedral phase. Consequently, this solid solution (β phase) regulates the composition of the alloys in the transformation to icosahedral ψ phase.⁽⁴⁾

The two compositions decagonal quasicrystalline alloys ($AlCuCo$ and $AlNiCo$) have its formation of a solid solution that derives from a common phase between both systems ($AlCo$) in that copper the compensation for the nickel. But, the decagonal phase is directly peritectic reaction with the liquid phase and the amount of aluminum present.

Most of these quasicrystalline alloys are metastable which become irreversibly at regular crystals after heat treatment or amorphous shape with small crystals of irregular shape. It has shown that almost crystalline phase formed in $Al_{62.2}Cu_{25.3}Fe_{12.5}$ conventionally solidified alloy is thermodynamically stable and do not suffer abrupt transformation phases (There may be small regions and amorphous intermetallic traces) until the melting point to 1135 K.⁽⁵⁾

However, the literature has shown that the quasicrystals icosahedron and decagonal phase is stable at high temperatures and transform the crystals and quasicrystalline phases low temperature.⁽⁶⁾

Studies were performed with the decagonal phase with the $AlCuCo$ alloying, have shown that type $CsCl$ cubic phase and bright dashes symmetrical around the decagonal phase; in sequential Al_2Cu intermetallic occurs with gray regions between the grains.⁽⁷⁾

The that diffuses scattering in decagonal quasicrystals spreads on the reciprocal two-dimensional planes and perpendicular to the axis of symmetry, while the icosahedral quasicrystalline alloy spreads over the three-dimensional reciprocal space.

This paper reports a study on the formation of icosahedral and decagonal phases, as well as oxidative behavior of each quasicrystal system $AlCuFe$ and $AlCoNi$. For this purpose we used the following techniques; X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS), calorimeter Differential Scanning (DSC) and Thermogravimetric Analysis (TG).

Materials and Experimental Procedures

Oxidation of the phase icosahedral and Decagonal

Oxygen generally exhibits different affinities with the intermetallic compound elements, leading to selective oxidation. However, oxygen reacts at the surface of quasicrystalline alloy metal oxides formed and the interface region beneath the oxide layer is preferably oxidized in the exhaust element.

The work of Werner et al⁽⁸⁾ showed the composition $AlCuFe$ oxidation starts with the growth of sequentially $\gamma-Al_2O_3$ are formed $\theta-Al_2O_3$, thereby have a strong nuclei of alumina $\alpha-Al_2O_3$ at the interface oxide / metal random manner through the copper layer growth; then processing occurs $\theta-Al_2O_3$ for $\alpha-Al_2O_3$, These steps occur at elevated temperatures.

In the oxygen quasicrystalline alloy reacts in quasicrystal surface forming metal oxides. The interface region beneath the oxide layer is preferably oxidized in the exhaust element. Thus the concentration is shifted out of the range of stability may occur phase quasicrystalline and a phase change.⁽⁹⁾

In the system of quasicrystalline alloys $AlNiCo$ has occurred in the formation of thin layers of oxides, by preference oxygen is bonded to aluminum, nickel

continue while the metallic state. However, the atomic structure of the oxide layer of the binary NiAl alloy (110) is complex has certain similarities with the hexagonal phases of aluminum oxide.

The composition of the aluminum oxide layer on the icosahedral phase to AlCuFe was observed by Chang et al.⁽¹⁰⁾ an aluminum enrichment tendency on the outer surface of quasicrystals. But the behavior of intermetallic components of quasicrystals in the presence of oxygen shows that the aluminum atoms move the mass to the surface. It is assumed to be due to the driving force provided by the exothermicity of the oxide being higher, the other alloy constituents as shown in figure 1 below.

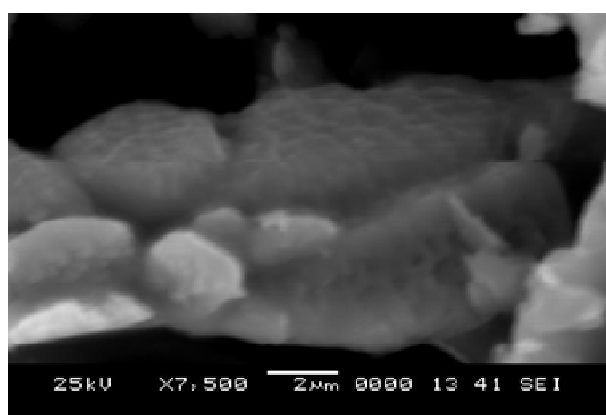


Figure 1. dense layer of alumina formed on the outermost surface of the AlCuFe alloy quasicrystalline.

According to S. Burckhardt et al, observed for the family of quasicrystal AlNiCo, the behavior of oxidation starting the technique of diffraction low-energy electrons (LEED); there is a decrease in quasicrystalline substrate, meaning a loss in intensity oxide structural layer. Completing this way, the oxide layer on the surface amorphous and crystalline continuously grows throughout the oxidation process. Thus, the points of electron diffraction correspond to aluminum to form film, which deposits on the surface decagonal $Al_{65}Ni_{15}Co_{20}$. Resulting in growth of the size of the field oxide layer between the quasicrystal/crystal interface.⁽¹¹⁾

According to studies of oxidation Dubois and his collaborators, the quasicrystalline alloy in the i-AlCuFe phase, the initial oxide layer is thin at a

temperature of about 670°C. The aluminum oxidation was still observed even after a long exposure to more than Langmuir 7000. The existence of a dense layer with passivating and inhibiting property copper

oxidation, as it influences the properties of adhesion/adsorption.⁽¹²⁾

Materials and Methods

The metallic powder aluminum, iron, copper, cobalt and nickel as its particle size, having a 99.9% purity, from Aldrich Chemical, wereweighed in the appropriate proportions composition ($Al_{62,2}Cu_{25,3}Fe_{12,5}$ e $Al_{65}Ni_{15}Co_{20}$), They were mechanically homogenized and compacted in the form of disc with a diameter of 10 mm and 2 mm thick in a hydraulic uniaxial press. Initially, it made a pre-pressing under full load of 5 ton for 3 minutes. The precursor alloys were obtained by direct fusion of compressed constituents. The fusion was carried out in arc furnace under controlled atmosphere of argon. The arc furnace is fed by a static rectified BALMER generator whose power is 22 kVA. For the identification of the phases, it used a Shimadzu XRD 6000 diffractometer using $CuK\alpha$ radiation with a wavelength of 1.5406 Å.

The measurements were taken for a wide range of diffraction angles (2θ) range from 20° to 120° angular pitch 0.05° and a timing point at 2.4 s. The analysis by SEM / EDS was performed with a Shimadzu SUPERSCAN SSX-550 with an acceleration voltage of 0.5 to 30kV with a 10V step, after the sample has been coated with a gold layer deposited under vacuum in order to improve contrast.

The thermo gravimetric analysis (TGA) and differential scanning calorimeter (DSC) were made on a Perkin Elmer TG 6000 STA TE equipment. Samples of quasicrystalline alloys were properly weighed and subjected to heating from ambient temperature (approximately 27°C) to 750°C at a heating rate of 10°C/ min under an inert atmosphere with nitrogen flow of 50 ml/ min.

Results and Discussion

X-ray diffractogram

Both Figures 2 and 3 show the XRD patterns of the compositions of quasicrystals ($Al_{62,2}Cu_{25,3}Fe_{12,5}$ and $Al_{65}Ni_{15}Co_{20}$), as is shown below respectively. The diffractogram 1 shows different layer phases present, is main peak in icosahedral phase and others to monoclinic phase $\lambda-Al_3Fe_4$, the other peaks is related to the cubic structure of the type of CsCl ($\beta-AlFe(Cu)$ or $\tau-AlCu(Fe)$), and $\theta-Al_2Cu$ tetragonal phases were detected. However, the crystalline phases are in equilibrium with each other; but between the

crystalline phase's ω and β icosahedral phase has a great similarity. The coordination of Fe atoms is very similar in both structures, the more this phase appears in three fields. Interestingly, the quasicrystalline phase always coexists with β -crystalline.⁽¹³⁾ The equilibrium solidification for this transformation is the primary forming λ - $\text{Al}_{13}\text{Fe}_4$ phase in the liquid phase giving rise to more of β phase by a peritectic reaction and finally the phase-i is formed by one second peritectic reaction of the type $L + \beta + \lambda \rightarrow \text{phase-i}$.⁽¹⁴⁾

The diffraction X-ray from the sample AlNiCo show three decagonal phases. In this structure is a nearly periodic symmetry plane along the direction perpendicular to these planes. Subsequently Al-Co, Al-Ni and Ni-Al-Co; and the periodicities of approximately 0,4, 0,8, 1,2 and 1,6nm in a specific direction, meaning that there was a rapid solidification in this composition. To identify $\text{Al}_{65}\text{Ni}_{15}\text{Co}_{20}$ alloy formed plans for five reciprocal vectors corresponding to five adjacent planes $\frac{2\pi}{5}$ with an angle of six vectors perpendicular to the other planes. All diffraction peaks observed in the quasicrystal may be indexed in a single decagonal phase and no diffraction peaks corresponding to other phases. The value ΔQ of the decagonal alloys are smaller than icosahedra's alloys, the difference is particularly remarkable in the main peak reflection, $Q \cong 30.0\text{nm}^{-1}$ indicating that the characteristic magnitude of the voltage to the phason of quasicrystalline structure is considerably lower for decagonal phase even after rapid solidification.⁽¹⁵⁾

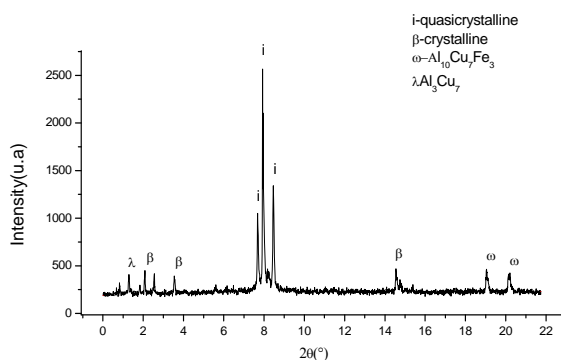


Figure 2. XRD pattern of quasicrystalline alloy $\text{Al}_{62.2}\text{Cu}_{25.3}\text{Fe}_{12.5}$

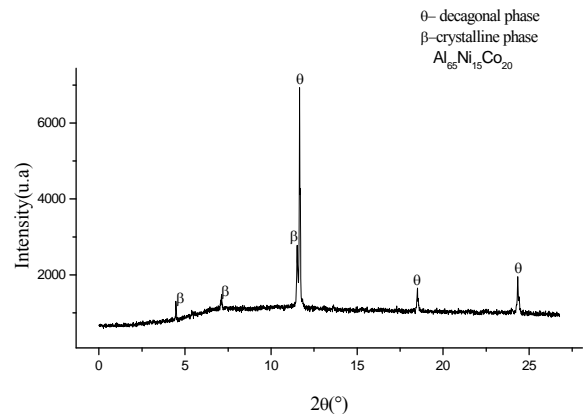


Figure 3. X-ray diffractogram (XRD) of the sample $\text{Al}_{65}\text{Ni}_{15}\text{Co}_{20}$ melt 900°C .

Scanning electron microscopy and EDS

In this electron microscopy analysis scan has as objective to verify surface morphology of the two quasicrystalline alloys; More EDS spectrum of the sample. EDS peaks correspond to the different elements present in the samples that were obtained by the arc furnace. This surface of Figure 4 of $\text{Al}_{62.2}\text{Cu}_{25.3}\text{Fe}_{12.5}$ noted faceted, with disagreements format "cauliflower". It is seen that the nucleation of β phase can occur in which sites, platelet grow from this interface to the core of small crystals nodules form on the icosahedral phase. But, it is appropriate to say that the refinement in grain boundaries and /or grain surface with protrusions. In this initial phase oxidation observes a surface topography mode, to form a thin oxide layer with a thickness in micrometers (μm). After a period of two hours lumps are observed irregularly distributed on the grain surface.

We verified this second SEM image, corresponding to Figure 5 of the quasicrystal $\text{Al}_{65}\text{Ni}_{15}\text{Co}_{20}$ oxidative behavior surface 10 times the symmetry of quasicrystalline alloy. For this D-AlNiCo composition is seen that the oxidation occurs in consequence, the dissociative adsorption of oxygen, which leads to a very thin layer of chemically oxygen. Similarly, the previous image AlCuFe alloy, the formation of nodules islands, which form a layer of aluminum oxide with a thickness of 5\AA consisting of five different areas of hexagonal structure that are placed in the same azimuthal orientation on the surface.⁽¹⁶⁾

From Spectroscopy analysis of excited electrons by X-ray (LEED) Parka et al.⁽¹⁷⁾ observed a tendency of aluminum enrichment in the outermost surface of quasicrystalline alloys. More is initially chemisorption

of oxygen; secondly forming an oxide thin film. The growth of this film exhibits reach saturation in about 50 L. This is clearly saturation kinetics nature since once few monolayers of oxide are formed, O_2 chemisorption rate and ionic transport through the oxide film is extremely low in ambient temperature.

Composition analysis with an EDS as shown in Figure 6 shows that the regions are composed mainly of Cu, Fe, O and a small amount of Al that can complex with Cu, Al_2O_3 , $AlFe_3$, Al_3Cu , Cu_2O and Fe_3O_4 or $CuFeO_2$ since the phases present in the transformation is due to the strong presence of Fe conduction electrons and Cu which are protected by the thin layer of aluminum oxide which enables the peritectic reaction between the phases of $\gamma-Al_{13}Fe_4$ liquid and to form the highly dense phase $\omega-A_{17}Cu_2Fe_1$. The nodules represent β phase droplets formed mainly by surface tension of liquid (saturated caused by solid state reaction) in a bubble or hollow retraction (cavity) solidified in its original liquid form.

Therefore EDS can be seen in the spectrum of Figure 7, a more intense peak showing the greatest concentration of aluminum, this is due to higher amount of cobalt in nickel ratio in the decagonal phase. It must be said, the ternary composition $AlNiCo$ in decagonal phase occurs by substitution of Co and Ni, comes a vacancy formation in quasicrystalline alloy structure; this produces a disorder of the type of solid solution. The diffuse scattering is often observed in the XRD patterns in the decagonal phase. However, it should be noted that the portion contains higher amount of aluminum is from a continuous band solidly solution- β of isostructural $AlCo$ and $AlNi$. This decagonal phase is formed along on $Al_{73}Co_{27}-Al_{69}Ni_{31}$. Soon, you can check in the EDS spectrum is the second highest peak of the cobalt atom (Co). In decagonal phase quasicrystal presents stable structural variations; we noted the part with cobalt wealth is due to another structure similar to that of $Al-Co-Pd$ in ω -phase. In other compositions there are also other phases like decagonal orthorhombic.⁽¹⁸⁾ They were obtained by hybridization at a temperature of 800-900°C, while at higher temperatures, depending on the composition, and decagonal phase that was formed.



Figure 4. Morphology of showing nodules islands of quasicrystalline alloy $Al_{62,2}Cu_{25,3}Fe_{12,5}$,

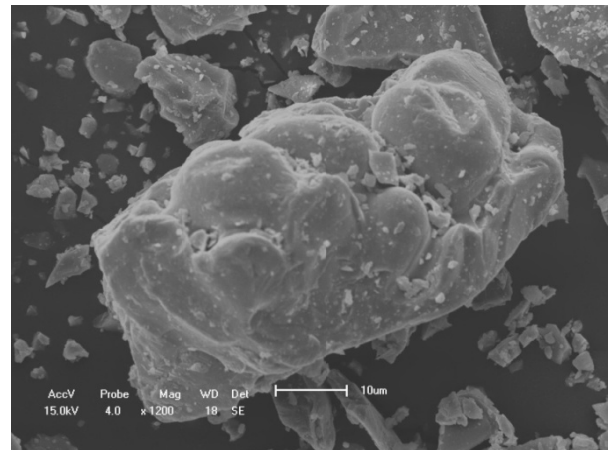


Figure 5. Structure of nodular decagonal phase of the quasicrystal $Al_{65}Ni_{15}Co_{20}$.

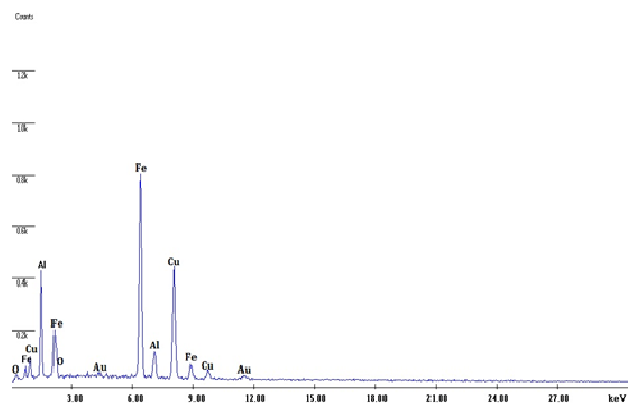


Figure 6. Analysis of EDS quasicrystalline alloy $Al_{62,2}Cu_{25,3}Fe_{12,5}$

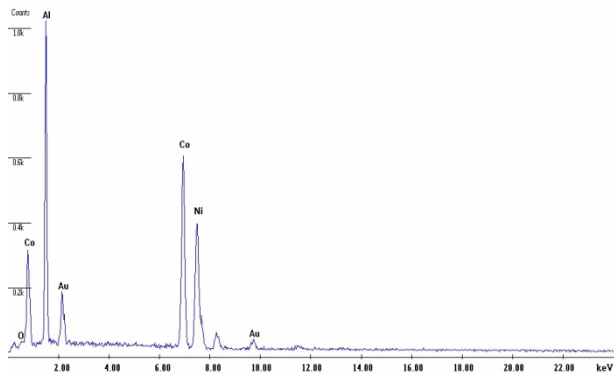


Figure 7. EDS representation of quasicrystalline alloy $\text{Al}_{65}\text{Ni}_{15}\text{Co}_{20}$

Thermal analysis DSC and TG

Figure 8 shows the DSC curve behavior as can be seen, it is well accentuated curvature at the temperature of 300°C to characterize an endothermic peak. Showing us that it is the aluminum melting point and pure copper in the formation of quasicrystalline alloy. This endothermic peak corresponds to melting of pure Al powder, Cu, following the dissolution of the liquid iron (Fe), after mechanical casting process. Therefore, at this phase in the quasicrystalline alloy AlCuFe is being processed by the arc furnace mergers, Al and Cu elements are coming into contact with one another in low-temperature is displayed on the graph.

So is revealing reaction is a eutectic melting, as is seen in the literature in binary phase diagram Al/Cu eutectic temperature. Underlining this stage of the mechanical process there is a predominance of aluminum the other copper and iron elements. However, we have an exothermic peak temperature of 800°C informing the occurrence of a series of solid crystalline phase transformation during heating for quasicrystalline phase. As is the homogenization of the alloy composition is gradually completed obtain the ω - $\text{Al}_7\text{Cu}_2\text{Fe}$ intermetallic to the main phase icosahedral.

As can be observed in the TG curve quasicrystalline alloy AlCuFe occurred small mass loss at temperatures as follows; The first mass loss was at 40°C for a percentage of 21.4%; Yet another loss occurred at 100°C for 21.6%; The third significant loss occurred at 410°C 21.9%; With increasing temperature of 700°C obtain a mass reduction of 22.6%. However, at lower temperatures thermal decomposition decreases with decreasing particle size, becoming a solid mass loss held between 45°C to 820°C. It can be said that the decomposition temperature provides

an enrichment of the element composing the quasicrystal and also a depletion of the alloying element in different regions of the grain.

Thus, the DSC curve for the training quasicrystal $\text{Al}_{65}\text{Ni}_{15}\text{Co}_{20}$ revealed similar factors with AlCuFe, but we note that the endothermic peak occurred at a transition temperature from 320°C to 400°C. In the literature it is considered that the phase boundaries are displaced from the use of mechanical synthesis process. Moreover, it is likely will change the decagonal face occurs in structural relaxation. Naturally, cooling and heating peaks composition AlNiCo alloys is reversible martensitic transformation. Other possible β -crystalline phases is coexists with the solid solution phase is of face-centered γ -cubic. However, the formation of crystalline domains AlNiCo thermal diffusion is the predominant effect, enabled only by Al atom being caused by plastic deformation in the decagonal phase.⁽¹⁹⁾

In referring to the TG curve of Figure 9 of the quasicrystal $\text{Al}_{65}\text{Ni}_{15}\text{Co}_{20}$ exhibited more significant mass loss of points in terms percentage/temperature AlCuFe. Initially, the first mass loss event has occurred at 35°C for 25.65% percentage. Then, the second weight loss occurred at a temperature of 395°C in 25.8%. The third and fourth points passed in certain temperatures 510°C with 25.9% and 700°C to 26.0%. The fifth point occurred at a temperature of 805°C to a mass loss of 26.2%. These weight losses shown in the TG curve has an influence on the oxidation of almost linear quasicrystal and the atmosphere of the environment.

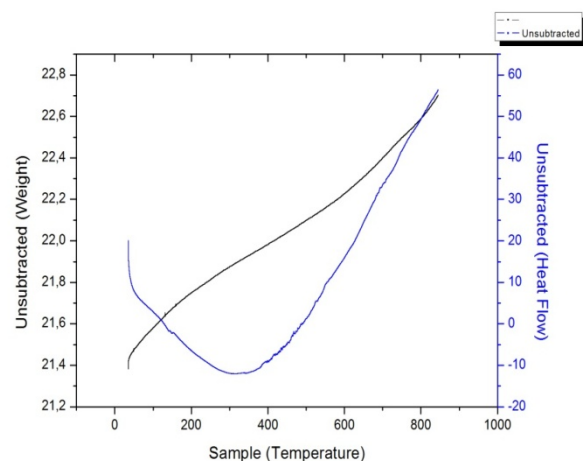


Figure 8. Representation of DSC and TG curves in the quasicrystal alloy $\text{Al}_{62.2}\text{Cu}_{25.3}\text{Fe}_{12.5}$.

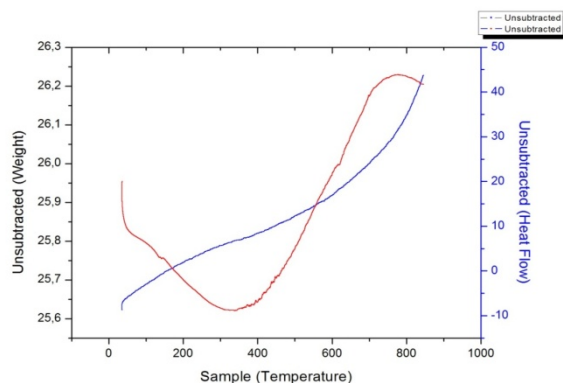


Figure 9. Representation of DSC and TG curves energy flow and mass of the quasicrystal $Al_{65}Ni_{15}Co_{20}$.

Conclusions

The main conclusions of the research are as follows:

1. The quasicrystalline phase becomes stable above $675^{\circ}C$ and the crystalline phase a solid solution which controls the formation of quasicrystalline phase.
2. In this decagonal alloying $Al_{65}Ni_{15}Co_{20}$ is the saturated type with vector perpendicular plane and rich of cobalt atom.
3. The effect of the high temperature causes variations in boundary defects of the quasicrystal $AlNiCo$ grains, these displacements are related to the temperature range default phason.
4. The $\lambda-Al_3Fe_4$ phase in the liquid phase giving rise to β -crystalline by peritectic reaction and form the i -phase which is comprised of a second type peritectic reaction $L + \beta + \lambda \rightarrow \text{phase- } i$.
5. The volume of distribution of the cobalt atom (Co) in quasicrystalline alloy $Al-Ni-Co$ got a reverse anisotropic diffusion behavior for self-diffusion along the periodic and aperiodic direction surface.
6. The existence of $\gamma-Al_2O_3$ provides the spinel formation on copper and iron oxidized forming CuO and FeO .
7. Oxidation of aluminum by changing concentration in the near-surface area induces phase transformation. Such transformations are abrupt since intermetallic phases are observed.
8. The decagonal quasicrystals oxidation effect of $Al-Ni-Co$ proceeds in oxygen consumption divided into two crucial stages; the first chemisorption have a rapid exposure to Langmuir 10 to 20; the following by a slower absorption presumably the formation of oxide thin film.

Acknowledgments

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