CHARACTERIZATIONS AND ELECTRON PARAMAGNETIC RESONANCE (EPR) QUASICRYSTALS IN AN ICOSEHEDRAL PHASE

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ABSTRACT

This article deals with a study on the icosahedral phase quasicrystal of Al₆Cu₆Fe₁₀ produced by casting in plasma induction furnace under an argon atmosphere to guarantee it’s homogeneously. But found in phase transformations from one phase to another, the structures formed by the quasicrystalline alloy having ordered icosahedral phase, leads to low electrical conductivity; but considering the location of weak electron-electron interactions. For microstructural characterization was made using the following techniques: X-ray Diffraction, Scanning Electron Microscopy, Energy Dispersive Spectroscopy of, DSC Thermal Analysis and Spectroscopy Electron Paramagnetic Resonance (EPR), where the spectrum obtained experimental EPR presents an amount of resonance g = 2.036. EPR spectrum provide a detailed view of magnetic equivalent sites to those of paramagnetic transition metal compositional variation in the alloy quasicrystalline and transition temperature effects on the magnetic spins in the sample ordering.

Keywords: EPR Spectrum; Quasicrystal; Microstructural Characterization; Icosahedral Phase.

1. INTRODUCTION

The quasicrystals are solid atomic structures with periodic and quasi crystallographic symmetry forbidden, since the physical and chemical properties are different from conventional crystalline solids, by presenting a long-range periodicity. The quasicrystalline alloy AlFeCu system and other families of quasicrystal have attracted considerable interest because of good physical and chemical properties, mechanical properties and tribological will know: a low electrical and thermal conductivity, good corrosion resistance and oxidation, high hardness and low friction coefficient. Investigative awakening in quasicrystal material comes from the thermodynamic instability, non-toxicity and low production cost of quasicrystalline alloy and great potential in various area of health, industry and technological field. Some important applications of this material under study are: Some important applications of this material under study are: photonics, electronic, magnetic, biomaterials and good hydrogen storers to be used in catalytic reactions.

The power system quasicrystalline Al-Cu-Fe has compositions in the range of 58-70% Al, 20-28% Fe and 10-14% Cu with, allowing the thermodynamic stability in quasicrystalline icosahedral phase. The transformation from the solid phase is due quasicrystalline (β crystalline, amorphous and icosahedral and present intermetallic). But it is emphasized the transition from one phase to another, there is presence of amorphous gamma this process occurs through nucleation from a single crystal of γ-Al₂O₃ as the core of a layer of amorphous alumina present in quasicrystal under normal temperature and pressure conditions. The preparation of the alloy Al-Cu-Fe, takes place through a thermal aluminum reaction as the reaction (1), this overheating is fusion or even the rapid solidification. They are used iron powders, copper and aluminum which are added to reduce the adiabatic combustion temperature and thereby avoid vaporization of the other components.

$$3\text{CuO} + 2\text{Al} \rightarrow \text{Al}_2\text{O}_3 + 3\text{Cu} + 1194,4\text{kJmol}^{-1} \quad (1)$$

Aluminum, copper and iron oxide powders were weighed according to the stoichiometry of equation (2) for the production of quasicrystalline materials Al-Cu-Fe with a nominal composition of Al₆Cu₆Fe₁₀.

$$7\text{Al}_{11} + 25\text{CuO} + 12\text{Fe} \rightarrow 8\text{Al}_2\text{O}_3 + \text{Al}_{16}\text{Cu}_{12}\text{Fe}_{12} \quad (2)$$

The icosahedral phase quasicrystalline present in the Al-Cu-Fe alloy is formed by peritectic solidification of crystalline and amorphous phases of high temperature react with a liquid phase.

In view of the formation of quasicrystalline phase from other phases occurred in icosahedral structure, the intermetallic compounds are related to the AlFeAlFeCu. The β-AlFe structure is a driving force in the formation of quasicrystalline phase AlFeCu. Therefore, these structures share similar properties and can say both are naturally brittle. Therefore, it is expected that the AlFeCu mechanism suffers from being fragile intermetallic system similar to AlFe.

When the quasicrystal is subjected to the heat treatment of 8 hours, no decrease in oxidation of the alloy, being able to obtain and observe the icosahedral structure as shown in Figure. 1.

![Figure 1. SEM image of the surface quasicrystalline alloy Al₆Cu₆Fe₁₀ in icosahedral phase.](image-url)

Conduction electrons is an important study in quasicrystals because a physical understanding of the electronic density of states. Therefore, another interesting aspect of that emerges from the location of the unpaired electron spins of the electrons present in quasicrystals in its magnetic moment contained in the transition metals (Mn, Ni, Co, Nb, and Fe) and rare earth metal atoms.

The interaction between the magnetic exchanges unpaired d f moments leads to magnetic ordering phenomena carrying a magnetic ordering of their spins or antiferromagnetic type at low temperatures.

However, to date the quasicrystalline alloy (Al-Mn-Si) and (Al-Mn-B-Pd) exhibited ferromagnetism by which were investigated by electron paramagnetic resonance, verified the existence of ferromagnetic resonance that is reported in ferromagnetic phase. This work has the purpose to study some techniques of...
characterization and magnetic effects through the use of electron paramagnetic resonance in the icosahedral phase of quasicrystal Al-Cu-Fe.

2. THEORY

2.1 EPR Spectroscopic Study of AlCuFe2 Quasicrystal in Non-magnetic Phase

The electrical resistivity of quasicrystals is several orders of magnitude, but in the transition metals is higher. Typical lower resistivity at room temperature are in the range $\rho=10^8-10^{10} \mu$Ωcm for quasicrystals decagonal and icosahedral with $\rho=10^{12} \mu$Ωcm, however, compared to the resistivity of aluminum metal which $\rho_{\text{Cu}}=0,3\mu$Ωcm. The resistivity of the quasicrystals and the remaining orders of magnitude lower than that seen in the insolation so that the quasicrystals can be generally considered as weak metals or semimetals.

Paramagnetic Resonance Spectroscopy metal gives rise to two reasons: The first is the fact that the intensity of the paramagnetic resonance spectroscopy line is proportional to the static spin susceptibility, the conduction electrons, it is of a type Pauli, their order of magnitude can be given by $^6$.

$$
\chi_s = \frac{g \mu_B J}{5 \mu_B T_F}
$$

(3)

An electron of an atom in the periodic quasi network then has a total angular momentum which is the sum of orbital angular momentum and orbital spin and spin in accordance with equation (4) below $^{10}$:

$$
\vec{J} = \vec{L} + \vec{S}
$$

(4)

The relation between the orbital and spin magnetic moments and their angular momentum is different, i.e. that the total magnetic moment, in general, is not parallel to the total angular momentum. Here $\mu_p$ the same relation holds for an atom as a whole, where an electron from an atom then has a total angular momentum. However the components and $\mu_p$ maintaining a proportional relationship which is expressed as:

$$
J_z = \hbar \mu_p
$$

(5)

In this case $j$ is the sum of the angular momentum of all its electrons and the quantum number can assume both integer values as semi-integer, where in unit steps and, is the Fermi temperature of the order of $10^9 \text{K}$ according to equation (6).

$$
T_F = \frac{E_F}{k_B}
$$

(6)

This susceptibility remains four orders of magnitude smaller than, the type of Cu sample. The paramagnetic susceptibility of localized electrons at electronic temperatures of liquid He to a comparable density of electron spins (since electrons are not located in a magnetically ordered state). The second reason for the weakness of the paramagnetic resonance spectroscopy signal is the surface that mediate the effect of conduction electrons with their.

Spins, where the microwave field penetrates the metal sample only to the radius of the layer of depth order given by,

$$
\delta = \frac{\rho}{\mu_0 B}
$$

(7), with $\mu$ is the magnetic permeability in vacuum.

The layer effect dramatically reduces the number of excited electrons which contribute to electron paramagnetic resonance signal. In the X-band electron paramagnetic resonance with the resonance frequency of free electrons $g = 2$ in $v = 9.6 \text{GHz}$ at a typical resistivity $\rho = 2.10^8 \mu$Ωcm quasicrystal in a sample which results in a depth of layer $\delta = 23 \mu$m $^{11}$. For electron paramagnetic resonance up field held in $v = 109 \text{GHz}$, the depth of the layer reduces the $\delta = 3 \mu$m. The microwave field can thus excite a small amount of conduction electrons with the surface layer of a single sample. The factor $g$ of conduction electrons in metals is generally not very different from the value of free electrons $g = 2$, the reason is that the exchange interaction between the conduction electrons fast moving average is zero and therefore unable to change the resonance frequency.

The small difference between actual values and the free electrons is of the order $g - g_p = 10^2$ normally found in metal; it is attributed to the spin-orbit coupling, which causes the electron energy dependent upon the relative orientation between its two angular momentum, orbital and spins $^{12}$. A related situation should be applied to quasicrystals as well. The average conduction electrons should be centered again towards the value of free electrons for the same reason. The spin-orbit coupling, however, deserves more attention in this study.

2.2 Electron Paramagnetic Resonance in Quasicrystals Magnetic

Some quasicrystals icosahedral systems such as; Al-Pd-Re, Al-Cu-Ni, Al-Cu-Ga, Al-Ba-Mo, Al-Cu-Mg, Al-Mn-Pd, Al-Co-Cu and Al-Pd-Ce are perfectly diamagnetic, others may contain paramagnetic centers in addition to itinerant magnetic moments of the conduction electrons. Examples thereof are transition metals containing icosahedral families Al-Pd-Mn, Al-V-Cu, Al-Fe-Ni, Pt-Al-Fe, Al-Co-Ni, Fe-Cr-Al and Cu-Al-Fe, where the Cr atoms, Co, Cu, Ni, Pt, Mn and Fe have unpaired electrons in their energy sublevel $d^{11}$.

In this case two types of electronic resonances can be observed in both the conduction electrons in the electron spin location in quasi periodic work.

The quasicrystalline alloy containing iron atom Hamiltonian of disturbed crystalline electric field is usually much greater than the spin-orbit coupling should result in small displacements $g$ of localized electron resonances. There is an important difference between the spin-orbit coupling induced displacements of the conduction electrons and localized moments. An electron located is moving in a spherical electrostatic potential with its electrons and the effect of the crystalline electric field meets the electronic orbital angular momentum, whereas it does not affect much the network own potential at the atomic level. The resulting displacement is therefore very small in quasi periodic quasicrystalline work.

A conduction electron on the other hand, moves in the crystal a total potential. For regular metals this potential has the periodicity of the structure. In quasicrystals the periodicity of the structure does not exist, so that significant variations in potential sites without transnational periodicity. This presents a distribution displacements $g$ which is not necessarily small. The conduction s electrons and localized spins are coupled by an exchange interaction of form $^{13}$,

$$
H_{\text{ex}} = \sum_{\alpha} \left( \vec{J}_1 \cdot \vec{S}_1 - \vec{J}_2 \cdot \vec{S}_2 \right)
$$

(7)

Here $J_1$ is the exchange coupling constant, $S_1 \cdot \vec{S}_1 - \vec{R}$ is the driving density of free electrons at one point $\vec{R}$, $S_1(\vec{R})$ it is the total spin of the d electron of the transition metal atom located at point $\vec{R}$, work and the sum is over all atoms of the magnetic structure of quasicrystalline superlattice. This interaction introduces a broadening and a shift of the resonance. However, as the two resonant frequencies are almost equal, the resonance of conduction electrons and localized spins are aligned at the same time have to meet the natural frequencies of the coupled spins.

The resonance frequency becomes a function of the frequencies of the two subsystems, the exchange parameter $J_1$ and the network spin relaxation damping representation of Spins Resonance spectroscopy of quasicrystal the sample occurred at the end of a resonant cavity. The quantity measured is the power absorbed by the sample from change when the external field is varied.

In this experiment the reflection was conducted in a band X (irradiation frequency 9.6 GHz, corresponding to the absorption center field 3400 G for $g = 2$ through a sample powder in quasicrystal Al-Cu-Fe, icosahedral phase. At room temperature until the liquid He, the spin resonance signal electrons can be detected just below 60 K in a relatively narrow line width, in the range of appeared $\Delta B = (4.5-5) G$ according to Figure 2.

Its significance factor $g$ varied in a small range to a low temperature of 4K its the driving density of free electrons at one point $\vec{R}$, $S_1(\vec{R})$ it is the total spin of the d electron of the transition metal atom located at point $\vec{R}$, work and the sum is over all atoms of the magnetic structure of quasicrystalline superlattice. This interaction introduces a broadening and a shift of the resonance. However, as the two resonant frequencies are almost equal, the resonance of conduction electrons and localized spins are aligned at the same time have to meet the natural frequencies of the coupled spins.

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carried out at a temperature below. The spectra obtained in this experiment did not appear clear or pure dispersion, but have a slight mixture of absorption. A weak signal with a narrow spectral width is observed near the position of free electrons in B = 3.8684 T, which is obviously the same seen in X band in Figure 2. In Figure 3 obtained by electron paramagnetic resonance shows the g factor for the icosahedral phase in quasicrystal Al-Cu-Fe where this shift is because of conduction electrons originating from the iron, because there was a slight variation for a low temperature 4 K, however, the intensity of the line increases by cooling the inverse of the Curie temperature 1/T.

Figure 2. Electron paramagnetic resonance signal representation of the icosahedral quasicrystal Al\textsubscript{4}Cu\textsubscript{3}Fe\textsubscript{12}, the 25 K. 2 (b) the intensity of the paramagnetic resonance signal of electrons. (Corresponding static susceptibility of electrons in the paramagnetic resonance) as a function of temperature.

The behavior of the icosahedral phase quasicrystal AlCuFe by some important magnetic properties by electron paramagnetic resonance, and the influence of the magnetic field and the Curie temperature factor 1/T, these factors magnetism quasicrystalline the alloy is crucial for applications some industrial sectors. The structures formed by quasicrystalline alloys have icosahedral phase orderly, provides low electrical conductivity, but is due to consider the location of weak electron-electron interactions. The microstructure effect on quasicrystal is observed by high polarization field, to go analyze heterogeneous spin caused by defects, dislocations and fluctuations of random magneto crystalline.

The Al\textsubscript{4}Cu\textsubscript{3}Fe\textsubscript{12} quasicrystal has localized magnetic moments on iron atoms (Fe) since only 10\textsuperscript{4} Fe atoms have centered moments. The resistivity of the icosahedral quasicrystal Al-Fe-Cu and Al-Pd-Mn are almost the same so that a similar concentration to conducton electrons must exist in both quasicrystals. Furthermore, both quasicrystal families are structurally almost identical.

3. MATERIALS AND METHODS

From the pre-established stoichiometry Al\textsubscript{62}Cu\textsubscript{25}Fe\textsubscript{12} were heavy aluminum powders (99.97%), iron (99.9%) and copper (99.9%) in an analytical balance, the Gehaka brand, AG-200. The alloy was obtained by melting the pure elements of air. The induction furnace was used with Argon atmosphere controlled at 5.0 with the objective of obtaining a good homogenization of quasicrystalline phase.

We used a high-frequency generator (40 kVA) POLITRON of manufacture. Each element that makes up the quasicrystalline alloy contains 10\textsuperscript{5} measurement procedure was performed on a Shimadzu balance. The training method was through solidification in cold hearth furnace to generate a heterogeneous alloy, being a common procedure a mixture of quasicrystalline phase to the crystalline phase.

To be a proportional increase of quasicrystalline phase in the sample, it is necessary to heat treatment, so that will favor the peritectic transformation of phases. This was accomplished using the heat treatment in a resistance furnace Nabertherm the mark, where the samples are maintained during the period of 8h and 24 h at a temperature of 1123 K. The diffraction of X-ray (XRD) was used to monitor the progress of the phases and stability of samples during casting. It was used for both a diffraction SIEMENS D5000 and is used CuK\textsubscript{α} radiation whose wavelength is λ=1,5406Å. To examine the morphology of the powders being quasicrystalline using a LEO Scanning Electron Microscope, Model 1430, OXFORD coupled to a probe. The samples after casting and were placed in catalytic tests dispersion in isopropyl alcohol solution. The energy dispersive spectroscopy (EDS) in the sample binds quasicrystalline Al\textsubscript{4}Cu\textsubscript{3}Fe\textsubscript{12} micrograph was obtained at an energy dispersive analysis after 8 hours heat treatment coupled with the SEM sample which has previously been metallized with gold (average thickness 12nm). The thermal properties of alloys were analyzed by Differential Scanning Calorimeter (DSC) at 10 °C min\textsuperscript{-1} heating rate under N\textsubscript{2} flow.

4. RESULTS AND DISCUSSION

4.1 X-ray Diffraction

The diffraction spectra of X-rays with the sample Al\textsubscript{4}Cu\textsubscript{3}Fe\textsubscript{12} stoichiometry is shown in Figure 4 respectively for the thermal treatment of 24 hours at 1123 K. This diffraction peak can identify the phases; the icosahedral quasicrystalline phase (i-phase) and tetragonal phases (θ-AlCu, ω-CuAl, and β-AlCu) and intermetallic (ω-Al\textsubscript{4}Cu\textsubscript{3}Fe\textsubscript{12}, λ-Al\textsubscript{4}Cu\textsubscript{3}Fe\textsubscript{12}, γ-Al\textsubscript{4}Cu\textsubscript{3}Fe\textsubscript{12}, θ-AlCu, ω-Al\textsubscript{4}Cu\textsubscript{3}Fe\textsubscript{12}, and ϕ-Al\textsubscript{4}Cu\textsubscript{3}Fe\textsubscript{12}).

These results also suggest that the β phase is directly formed from the liquid alloy. Moreover, the β phase transforms below 873 K to form the λ and θ stages, which are solid solutions induced by the solubility of Cu and Fe. The intensity of the peaks corresponding to the i-phase layer is higher than the peaks are specifically related to the β-phase.

However, Al\textsubscript{4}Cu\textsubscript{3}Fe\textsubscript{12} in the alloy melt, i-phase coexisting with a small amount of AlFe(Cu) solid solution. The composition of Al\textsubscript{4}Cu\textsubscript{3}Fe\textsubscript{12} have phases in balance with other crystalline phases such as β-Al\textsubscript{4}Cu\textsubscript{3}Fe\textsubscript{12}, γ-Al\textsubscript{4}Cu\textsubscript{3}Fe\textsubscript{12}, θ-AlCu, ω-Al\textsubscript{4}Cu\textsubscript{3}Fe\textsubscript{12} and ϕ-Al\textsubscript{4}Cu\textsubscript{3}Fe\textsubscript{12}. Among these phases ω crystalline shows a great similarity with the icosahedral ω phase. The coordination of Fe atoms is very similar in face-centered cubic structure with Cu being passivity with Al\textsubscript{2}O\textsubscript{3}. From the solidification path, the ω phase seems to be formed at equilibrium conditions with high thermodynamic stability, by a peritectic reaction from a crystalline and amorphous intermetallic phase due to phase transitions.
4.2 Scanning Electron Microscopy and EDS

Figure 5 and 6 are respectively represent the results obtained from samples of quasicrystals by Scanning Electron Microscopy and EDS spectrum post after being subjected to heat treatment with 8 hours.

The analysis Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) were used for sample quasicrystal and produced Figures 5 and 6 with the heat treatment of 8 hours. The image that the Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy spectrum (EDS), view the microstructure of quasicrystalline alloy with surface irregular crystals and geometric uniformity.

The phases present already been processed (β-crystalline, and intermetallic icosahedral), there are show the strong presence of Fe and Cu conduction electrons which are protected by the thin layer of aluminum oxide which enables the peritectic reaction between phases 16). In the spectrum of elemental analysis EDS, there is a greater prevalence of aluminum than the other elements (copper and iron) that makes up the quasicrystalline alloy. The existence of γ-Al_{2}O_{3} favors spinel formation on Al_{2}O_{3} oxidation in the presence of Cu or CuO. However, the possibility of formation of other oxides such as spine CuFe_{x}Al_{2-x}O_{4}, and CuAl_{2}O_{4} are complex to form a thin film, the passive quasicrystalline structure of the alloy.

4.3 Thermal Analysis DSC

Figure 7 shows the thermal behavior during non-isothermal heating made by Differential Scanning Calorimeter (DSC) conducted at a heating rate of 10° CMIM-1 for quasicrystalline alloy powder during the heating Al_{62}Cu_{25}Fe_{12}, 400-1473K, exothermic events occur are clearly defined and distinguished in a low temperature range of 773 K, however, relates to the formation of φ-AlFe_{3} and ω-Al_{7}Cu_{2}Fe phases, which represents a fusion event or dissolution of the phases (amorphous intermetallic) corresponds to merging with predominance of copper and iron. A more complex sequence of thermal events is observed above 773 K. The increased heating rate causes a moderate change (~293 K) of the phase transition temperatures towards higher values, otherwise the following thermal events does not change. The φ-AlFe_{3} phase is the main precursor of γ-phase (i-Al_{62}Cu_{25}Fe_{12}).

As the homogenization of the alloy composition is gradually completed, φ-Al_{62}Cu_{25}Fe_{12} makes if main constituent. The enthalpy of the minimum first peak is smaller than the other two peaks, indicating that the highest concentration of phases of φ-AlFe_{3} type (ω-Al_{7}Cu_{2}Fe_{3}) and (i-Al_{62}Cu_{25}Fe_{12}). This result indicates the need for a detailed analysis of the microstructural evolution and the formation and stability of constituent phases during non-isothermal heat treatments, because this enables rich oxide layer that is essential for catalytic purposes.
CONCLUSIONS

- The main conclusions of the study are as follows:
  - The icosahedral phase becomes stable above 948 K and the quasicrystal.
  - The sharp widening of the spectrum icosahedral quasicrystal as prepared by mechanical

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