

RESEARCH ARTICLE



ISSN: 2321-7758

## SYNTHESIS OF QUASICRYSTALLINE Al-Cr-Fe ALLOY BY MECHANICAL ALLOYING PROCESS AND ITS CHARACTERIZATION IN MAGNETIC APPLICATIONS

CH. VIJAYA KUMARI

Lecture in physics

S.S N College, Narasaraopet, Andhra Pradesh, India



### ABSTRACT

The quasicrystalline alloy contains the aluminium portion, exhibits particle dispersion on a nanoscale level providing high mechanical resistance values at operating temperatures and at high temperature. Due to its good structural and magnetic properties, and the interactions between the properties, the interest in obtaining this material. Quasicrystalline alloys generally show friction and wear resistance, good electrical and thermal insulators, are hard, used in photonic sensors and some quasicrystal formations are good hydrogen stores. In this research, to analyse the magnetic properties, she studied the use of mechanosynthesis to obtain the decagonal phases  $Al_{67}Cr_{23}Fe_9$ . Samples of quasicrystalline alloys were obtained by high-energy grinding with a 20:1 ball mass ratio, with a rotation of 300 rpm, in time intervals ranging from 1 to 20 hours in a Fritsch Pulverisette 5 planetary ball mill of the physical characterizations were made by SEM presented a microstructures with non-uniform symmetries and large nodules X-ray diffraction provides information on phase identification resulting from the alloy  $Al_{67}Cr_{23}Fe_9$  the formations of the decagonal and intermetallic and the presence of peaks of diffraction patterns typical of its crystallographic network. The magnetic measurements performed were based on the temperature  $M(T)$ , and also on the applied field  $M(H)$ . It can be concluded that it is possible to form quasicrystalline phases according to the grinding time and speed for alloy obtained by the mechanosynthesis process.

Keywords: Aluminium alloy, mechanosynthesis process, Structural and magnetic properties

©KY PUBLICATIONS

### 1.0 Introduction

Mechanosynthesis is a processing technique in the solid state, where products are formed by the transfer of energy from mechanical collisions in a high-energy mill. In the formation of alloys, the particles are repeatedly subjected to a fracture and welding process until they form the final phases<sup>1</sup>. The grinding for a "prolonged" time allows the

formation of phases with grains of nanometric dimensions that are of great importance for their applications in several areas. Quasicrystalline alloys have a special feature of high thermal stability and mechanical properties at elevated temperatures much higher than those of conventional alloys<sup>2</sup>. Various systems may have icosahedral quasicrystalline particles with spheroidal morphology surrounded by a centered phase cubic

aluminum phase, such as Al-Fe-Cr-Ti and Al-V/Mn-Ti/Fe/Co/Ni- Ln<sup>3</sup>. The alloys that fit into these systems generally contain Al concentrations in the range of 75 to 95 (% at.). The mixed microstructure that is observed in these alloys is formed in a single solidification stage, with the icosahedral phase precipitating as the primary phase from the liquid, followed by aluminum precipitation, from the remaining liquid<sup>4</sup>.

The quasicrystals formed are mostly stable and are aluminum-based, normally the equilibrium phases can normally be predicted from the phase equilibrium diagram. In addition, quasicrystalline alloys that are metastable are irreversibly transformed into regular crystals after heat treatment or in amorphous forms with small irregularly shaped crystals<sup>5</sup>. Quasicrystalline powders are thermodynamically stable at temperatures above 800 ° C under an inert gas atmosphere and above 500 ° C in air. On the other hand, the effects of defects possibly generated during mechanical grinding and the influence of the particle size of the powders on the stability of the material<sup>6</sup>. That a quasicrystalline decagonal phase. Al<sub>65</sub>Cu<sub>20</sub>Co<sub>15</sub>, in powder, undergoes transformation to a nano-crystalline phase when ground for 30 h<sup>7</sup>.

However, the magnetic property of the quasicrystalline must be emphasized in the icosahedral phase and the stability of quasicrystalline alloys is being investigated in terms of the linear relationship between the magnetic susceptibility and the square temperature function. However, suggesting a particularly accentuated structure with depression in the density of the electronic states in Fermi energy; the dependence on the temperature raised to the square can be interpreted, as a result of the temperature dependence of Pauli's paramagnetic<sup>8</sup>. It should be said, the low chemical reactivity of quasicrystals is attributed to the presence of pseudo gaps, which is a reduction in the electronic density of states on the Fermi

surface. But, it is low reactivity with oxygen, it can be compared with the reactivity of the quasicrystalline alloy of Al-Cu-Fe with its crystalline analogues. The oxidation of alloys subjected to high temperatures is quite complex, since several technical activation processes are involved<sup>9</sup>. The development of this research work has as main focus the use of the high energy grinding process to obtain quasicrystalline material (Al-Cr-Fe) and the effects of processing and influences on the physical and chemical properties of the material under study. For a better understanding of the proposed theme, this work focused on synthesis and characterization of Al-Cr-Fe in stoichiometric combination.

## 2.0 Materials and Method

**2.1 Methods:** The quasicrystalline alloys prepared were characterized by Technics; XRD, SEM, EDAX and Vibrating Sample Magnetometers to assess their physical and magnetic properties.

Mechanical Alloying this process obtained a small amount of alloys and Al<sub>67</sub>Cr<sub>23</sub>Fe<sub>9</sub> in which, study the evolution of milling times (1, 5, 10, 20h) in this material.

**2.2 Preparation of the quasicrystalline alloy (Al-Cr-Fe) by Mechanical alloying:** The quasicrystal elemental powders with a purity of 99.99% for each element which constitutes the compositions were weighed on a precision balance to give the nominal compositions. The precursors were placed in steel jars, and held washes in argon atmosphere condition. So, to have accurate information of the total mass of the alloy powder has the relationship ball/ powder, as determined by 20: 1, correctly indicating the total sample weight, 20g; the ball sizes used in this procedure experimental were 10mm. In quasicrystals two materials, a mill used with discretion following a planetary mill, Fritsch Pulverisette model P5.

Table 1: Parameters variables used in the execution of grindings for the selected alloys.

Composition of alloys	Mill	Size of Ball (mm)	Speed (rpm)	Relationship Ball / powder	Mass of powders (g)	Vacuum (mbar)
Al-Cr-Fe	Planetarium Fritsch Pulverisette P5	12	250	25.2	22	6x10 <sup>2</sup>

In this experimental preparation of quasicrystalline alloy (Al-Cr-Fe) was a repetition of an identical sequences; time in minutes mill ( $t_m = 45$  min), followed by the stop time ( $t_p = 5.0$  min). The time of each grinding corresponds to the following equation: ( $n \times TM$ ) so we have the sequence to other milling times ranging from 1, 5, 10 & 20 hours.

Both alloys processed by mills did not use any lubricant or PCA process control agent (Process Control Agent).

### 3.0 Results and Discussion

The present work we have quasicrystalline alloy (Al-Cr-Fe) and subsequent characterization for identify the formed phase. For this purpose we organized a chronological sequence presented follow:

- X-ray diffraction patterns: In order to identify the phase formation, secondary phases compounds and some behaviours typical of mechanical alloying process.
- Scanning electron microscopy (SEM): For morphological characterization and study the possible contamination with jars and balls during the milling process.
- Energy dispersive spectroscopy X-ray (EDS): Identify the quantify the most predominate elements of quasicrystalline alloy.
- Vibrating sample magnetometer (VSM): For the magnetic basic study in order to show if some unexpected properties are presents in the obtained material.

**3.1 X-ray diffraction patterns of the prepared alloy:** The results of X-ray diffractogram of the samples with  $Al_{67}Cr_{23}Fe_9$  is shown in Figure 2 for 20 hours milling time. In this work it was possible to observe the influence of milling parameters on the formation phase of quasicrystal. The structural evolution, during the milling process was observed in function of time (0-5h) indicating a significant broadening of the diffraction peaks for  $Al_{67}Cr_{23}Fe_9$  alloy.

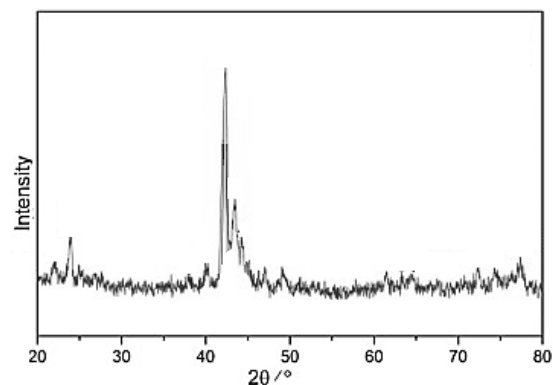
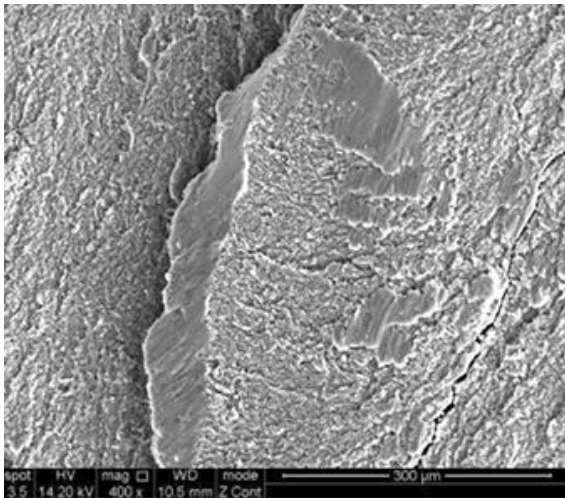


Figure 2: XRD Image for the sample  $Al_{67}Cr_{23}Fe_9$  for 20 hours of grinding.

In this case  $Al_{67}Cr_{23}Fe_9$  notoriously initially observed also in the second diffraction pattern in Figure 2 the presence of intermetallic phase  $Al_3Fe$  and a solution of Al and Cr based precursors. However, the sample milled for 20h observes the icosahedral phase due to the presence of chromium<sup>10</sup>.

### 3.2 Scanning Electron Microscopy (SEM)

The image presented in quasicrystalline alloy  $Al_{67}Cr_{23}Fe_9$  show microstructure with irregular shapes and porosity as the respective figure 3 below.

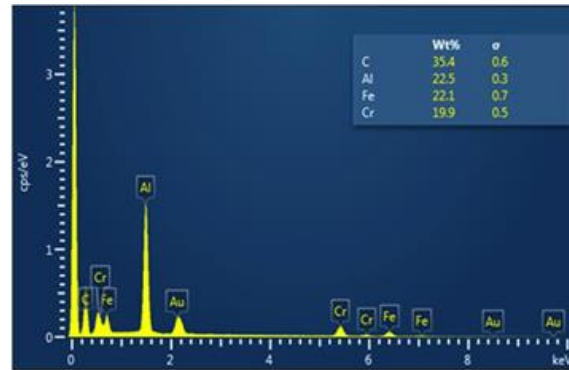


**Figure 3:** Micrograph of Sample  $Al_{67}Cr_{23}Fe_9$  dense layer of alumina.

The corresponding micrograph Figure 3, Sample Al-Cr-Fe is observed with longer grinding time in 10 hours, this microstructures presenting with irregular symmetry and pointed a little uniform with disagreements in their relief, that become structures crystals. This probably indicates that a large volume fraction of the solid particles collide with each other on the metal surface during the grinding process<sup>11</sup>. In the figure 3, the beginning of the thin layer of aluminum oxide with pores is white points, meaning the aluminum oxidation in the alloy in the form of aluminum oxide  $Al_2O_3$ . According to Chang et al observed the tendency of aluminum enrichment in the outermost surface quasicrystalline alloy. The behavior of intermetallic constituents of the icosahedral quasicrystal and decagonal phase, 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 which is higher than that of other alloy constituents<sup>12</sup>.

### 3.3 Energy Dispersive X-ray Spectroscopy (EDAX):

The EDAX spectrum of quasicrystalline alloy  $Al_{67}Cr_{23}Fe_9$  as lying below (Figure 4).



**Figure 4:** EDAX analysis of quasicrystalline alloy  $Al_{67}Cr_{23}Fe_9$

Composition analysis with an EDAX as shown in Figure 4, the observed regions have been composed mainly of Fe, Cr, O, and a larger amount of Al, they can complex with Cr,  $Al_2O_3$ ,  $AlFe_3$ ,  $Al_3Cr$ , and  $Fe_3O_4$  since the phases present in the transformation is due to the strong presence of conduction electrons of Fe and Cr are protected by the thin layer of aluminum oxide which enables the peritectic reaction between the phases of  $\gamma-Al_3Fe_4$  liquid and to form the highly dense phase  $\omega-Al_7Cr_2Fe_1$ . Aluminum is more prevalent than the other elements (copper, chromium, manganese and iron) in exact proportions<sup>13</sup>, since the existence of  $\gamma-Al_2O_3$  favor the formation of spinel on the copper in the alloy  $AlFeCr$ , and oxidized iron  $AlCrFe$  forming  $FeO$ .

### 3.4 Vibrating Sample Magnetometer (VSM)-Magnetization measures in quasicrystalline alloy

Magnetization measurements as a function of the applied field were made in  $Al_{67}Cr_{23}Fe_9$  sample. These graphics are represented respectively in the figure 5. Through the hysteresis curves we obtained magnetic characteristics of the coercive field, the remanence magnetization and saturation magnetization. We can see for the compound obtained here, that alloy is magnetically soft and coercivity and remanence have low values.

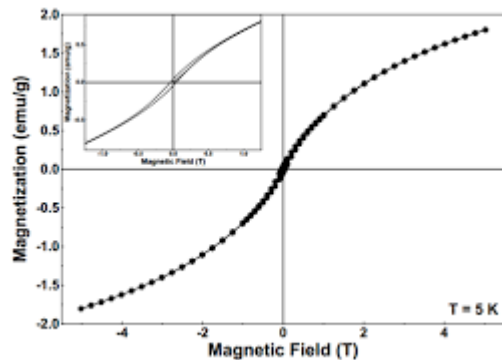


Figure 5. Hysteresis curve as a function of the field Al-Cr-Fe

Magnetic measurements above showed that our material have the expected behaviours for a quasicrystalline alloy. In other hand it is not sufficient to conclude many aspects about the magnetic internal conditions typical to the obtained material. In future experimental experiences we will use electron paramagnetic resonance in order to identify the role of the present atoms in the alloy showed here and Mossbauer spectroscopy to the iron atoms information<sup>14</sup>. Some experiments including the thermal dependence for the magnetic parameters showed above will be executed in order to understand the magnetic characteristic of our samples.

#### 4.0 Conclusion

The present work presented the quasicrystalline alloy of Al-Cr-Fe obtained by mechanical alloying. The initial characterization finding some magnetic and structural behaviors devolved important conclusions are it is possible to obtain the quasicrystalline alloys based in Al using mechanical milling by short milling times. Obtaining the system Al-Cr-Fe mechanical alloying allow a large amount of already quasi crystal powder form. In transitions from crystalline phases, intermetallic, icosahedral and decagonal possibly occur stability of the stages in the milling process and the grinding time influences the average grain size is about 19nm with 5 hours of milling. The XRD shows the presence of the main phases corresponding to each alloy quasicrystalline decagonal Al-Cr-Fe. The

images of SEM of quasicrystalline alloy are displaying formations of large nodules with spherical shape of the resulting intermetallic phases with oxide regions. The EDAX showing the peaks with greater intensity peaks, meaning the highest prevalence and amount of aluminium element to the other constituent atoms. The VSM showed basic properties characteristic of the above mentioned material. But it is not sufficient the magnetization curves for inferring detailed aspects of the material.

#### References

- 1 Suryanarayana, C. & Ivanov, Eugene & Boldyrev, V.V. (2001). The Science and Technology of Mechanical Alloying. *Materials Science and Engineering: A*. 304. 151-158. 10.1016/S0921-5093(00)01465-9.
- 2 Salimon AI, Korsunsky AM, Shelekhov EV, Sviridova TA, Kaloshkin SD, Tchrdyntsev VS et al. Crystallochemical aspects of solid state reactions in mechanically alloyed Al-Cu-Fe quasicrystalline. *Acta materialia*. 2001; 49(10):1821-1833
- 3 Dám, Karel & Průša, Filip & Vojtěch, Dalibor. (2014). Structural and mechanical characteristics of the Al-23Si-8Fe-5Mn alloy prepared by combination of centrifugal spraying and hot die forging. *Materials Science and Engineering A*. 610. 197-202. 10.1016/j.msea.2014.05.045.
- 4 Inoue, Akihisa & Kimura, Hisamichi. (2000). High-strength aluminum alloys containing nanoquasicrystalline particles. *Materials Science and Engineering: A*. 286. 1-10. 10.1016/S0921-5093(00)00656-0.
- 5 Orozco-González, Pilar & Castro-Román, Manuel & Muñoz-Valdez, R. & Luna-Álvarez, S. & Equihua-Guillén, F. & Hernández-Rodríguez, A. & Baltazar-Hernandez, Víctor & Alvarado-Hernández, F.. (2016). Formation and crystal structure of the  $\tau$  phase in the Al-Fe-Mn-Si system. *Materials Letters*. 180. 10.1016/j.matlet.2016.05.139.
- 6 Travessa, Dilermando Nagle, Cardoso, Kátia Regina, Wolf, Witor, Jorge Junior, Alberto

- Moreira, & Botta, Walter José. (2012). The formation of quasicrystal phase in Al-Cu-Fe system by mechanical alloying. *Materials Research*, 15(5), 749-752. <https://dx.doi.org/10.1590/S1516-14392012005000046>
- 7 Proveti JR, Larica C and Passamani EC. Structural properties and phase transformation in mechanically alloyed Al/Cu/Fe system. *Journal of Physics D: Applied Physics*. 2003; 36(7):798-804.
- 8 Barua P, Murty BS, Mathur BK and Srinivas V. Nanostructured icosahedral phase formation in Al<sub>70</sub>Cu<sub>20</sub>Fe<sub>10</sub> by mechanical alloying: Comprehensive study. *Journal of Applied Physics*. 2002; 91(8):5353-5359.
- 9 Murty BS, Barua P, Srinivas V, Schurack F and Eckert J. Synthesis of (Al<sub>65</sub>Cu<sub>20</sub>Fe<sub>15</sub>)<sub>100-X</sub>Si<sub>X</sub> quasicrystalline alloys by mechanical alloying. *Journal of Non-Crystalline Solids*. 2004; 334 & 335:44-47.
- 10 C. Dong, Li-M Zhang, Q.G. Zhou, H.C. Zhang J.M. Dubois Q H Zhang, Y.C. Fu, F.Z. He, and F. Ge, *Bull. Mater Science* 22, 465 (1999) 62(1):917-923.
- 11 Yadav, Thakur & Mukhopadhyay, N.K. & Srivastava, O.N.. (2014). Synthesis of Nano-Decagonal Quasicrystalline Material by Mechanical Alloying. *Advanced Science Letters*. 20. 10.1166/asl.2014.5494.
- 12 Enayati, Mohammad Hossein & Mohamed, Farghalli. (2014). Application of mechanical alloying/milling for synthesis of nanocrystalline and amorphous materials. *International Materials Reviews*. 59. 394-416. 10.1179/1743280414Y.0000000036.
- 13 Srivastava,V.C.;Huttunen Aarivirta,E.; Cui,C.; Uhlenwinkel,V.; Schulz,A.; Mukhopadhyay, N.K. Bulk synthesis by spray forming of Al-Cu-Fe and Al-Cu-Fe-Sn alloys containing a quasicrystalline phase. *Journal of Alloys and Compounds* 597, pp.258-268, 2014
- 14 B. Chitsazan, H. Shokrollahi, A. Behvandi, and M. Ghaffari, *Journal of Magnetism and Magnetic Materials* 323, 1128 (2011)