International Research Journal of Engineering and Technology (IRJET) e-ISSN: 2395-0056
Volume: 05 Issue: 09 | Sep 2018 www.irjet.net p-ISSN: 2395-0072

Synthesis and Characterization of Al-Cu-Mn Quasicrystal

Gururaj Hattaraki[1], Dr. B. Ravindra[2], H S Manjunath[3], Siddesh kumar C D[4]

[1]*M.Tech Scholar Dr. Ambedkar Institute of Technology Bangalore*
[2]*HOD & Prof. Dr. Ambedkar Institute of Technology Bangalore*
[3]Assistant *Prof. Dr Ambedkar Institute of Technology Bangalore*
[4] *Atria Institute of Technology Bangalore*

Abstract - *A quasiperiodic crystal, or quasicrystal, is a structure that is ordered but not periodic. A quasicrystalline pattern can continuously fill all available space, but it lacks translational symmetry. Quasicrystals are aperiodic crystals these are also called incommensurate structures. These crystals have 5 fold symmetry and can be described by pendrostyllic and Fibonacci numbers. Quasi-crystalline Al_{65} Cu_{20} Mn_{15} powder has been produced by mechanical alloying from crystalline elemental powders. The starting material consisted of elemental crystalline powders with a particle size smaller than 150 µm. Al-Cu-Mn quasicrystals will be synthesis by planetary ball milling. The ball to powder weight ratio 5:1 .During milling after 20 hours of milling we observed that the transformation from a mixture of elemental crystalline material to quasi crystalline alloy . The alloying process has been monitored by x-ray diffraction. The results will be analyzed and made it the report.*

Key Words: Quasicrystal; Powered Metallurgy; Materials; Milling of Al_{65} Cu_{20} Mn_{15}

1. INTRODUCTION

Quasi-crystalline Al_{65} Cu_{20} Mn_{15} fine particles has been produced by mechanical alloying from crystal-like elemental fine particles. The alloying process has been observed by x-ray diffraction, and the resultant invention has been characterized by transmission electron microscopy. The quasi-crystalline phase forms after about 125 hrs of milling. The crystallization temperature and enthalpy have been determined by differential scanning calorimetry. The results are compared with data for melt-spun material

1.1 Preparation methods of quasicrystals

1. The melt spinning technique 2. Other production technique for metastable quasicrystals 3. Conventional casting

All the production methods relevant to metastable alloys and glasses have been also applied to quasicrystals. They are all based on disorder generation at the atomic level. This is generally done by a solid state reaction. A typical method is the multilayer deposition technique in which alternating layers of Al and Mn are deposited on a substrate, the thickness being of the order of 1000 A0. Once the multilayer with the right average composition is obtained, the sample is bombarded by high-energy ions of inert gases (e.g. Xe2+).An Amorphous quasicrystalline or crystalline state is obtained, depending on the energy of the ions and on the sample temperature. Here disorder is introduced by the kinetic energy of the ions (like a ball in a bowling game) and is also driven by the temperature of the sample (the Synthesis and Characterization of Al-Cu-Mn Quasicrystal atoms become more mobile as the temperature increases). The samples obtained are quite small (2 X 2 mm2 and 1000 A0 thick) and are ideal for electron microscopy studies. Using this technique, single quasicrystalline phases can be obtained in the Al Mn system, allowing the study of the various phase transformations between quasicrystalline, amorphous, and crystalline states. The AI Mn quasicrystalline state can also form by direct ion implantation of Mn in an oriented Al matrix. Mechanical alloying (also called ball milling) can also produce amorphous or crystalline states. Powders of the different elements are alloyed by the kinetic energy of balls vibrating in a steel container. For ex-Eckert et al (1) obtained amorphous or quasicrystalline samples in the AlCuMn system. Apart from solid state reaction preparation, two other techniques should be considered: the evaporation technique and the laser or electron melting of thin layers. In the former method, a 'fog' of small droplets of liquid alloy is p reduced, and quenched. The 'smoke particles' present interesting external shapes and structures, with typical sizes in the range 500 to 3000 A0. Ishimasa et al. Have prepared dodecagonal quasicrystalline phase in the Ni Cr System using laser or electron melting of a small sample of thin layers; quenching results from radiative effects and the thermal capacity of the rest of the sample.

1.2 Method of production of quasicrystal by planetary ball milling

A ball mill is a type of grinder used to grind and blend materials for use in mineral dressing processes, paints, pyrotechnics, ceramics and selective laser sintering. A ball mill works on the principle of impact and attrition: size reduction is done by impact as the balls drop from near the top of the shell. A planetary ball milling consists of at least ore grinding jar which is arranged eccentrically on a sun wheel .The direction of movement of sun wheel is opposite to that of the grinding jar The grinding ball in the grinding jar are subjected to the superimposed rotational movements of the corislis forces This measurement and others are deliberate, using specifications, the different in speed between the ball and grinding jars produces in interaction between frictional and impact force ,which releases high dynamic engine The interplay between these force produce the high and very effective degree of size reduction of the planetary ball mill. The extremely high centrifugal force of

planetary ball mills results in very high pulverization energy and therefore short grinding times.

Fig-1: planetary ball milling apparatus

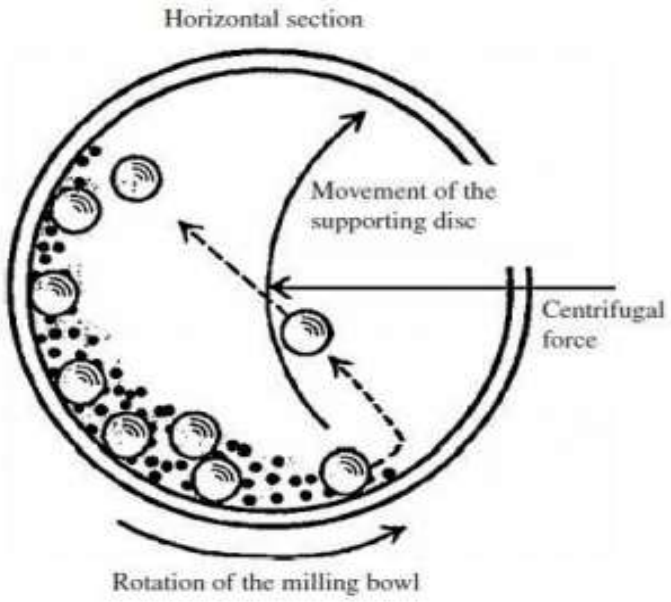


Fig-2: Pulverization mixing

Major parameters for ball milling

1. Temperature
2. Size and numbers of balls
3. Nature of ball
4. Rotation of speed

2. CHARACTERIZATION OF QUASICRYSTALS

X-RAY DIFFRACTION

x-ray diffraction is a tool used for identifying the atomic and molecular structure of a crystals ,in which the crystalline atoms cause a beam of incident X-rays to diffract into many specific direction. By measuring the angles and intensities of these diffracted beams , a crystallographer can produce a three-dimensional picture of the density of electron with in the crystals .From this electron density ,the mean position of the atoms in the crystal can be determined , as well as their chemical bonds ,their disorder and various information. In a single-crystal X-ray diffraction measurements, a crystals is mounted on a goniometer. The goniometer is used to position the crystals at selected orientations .The crystals Is bombarded with a finely focused monochromatic beam of X-rays , producing a diffraction pattern of regularly spaced spots known as reflection .The two-dimensional images taken at different rotation are converted into a three-dimensional model of the density of electron with in the crystal using the mathematical method of Fourier transforms ,combined with chemical data known for the sample.

The X-Ray Diffraction patterns are the signature of the elements which uses BRAGG'S Law. It is the necessary condition X-Ray Diffraction, however it is not sufficient The BRAGG'S Law is as follows

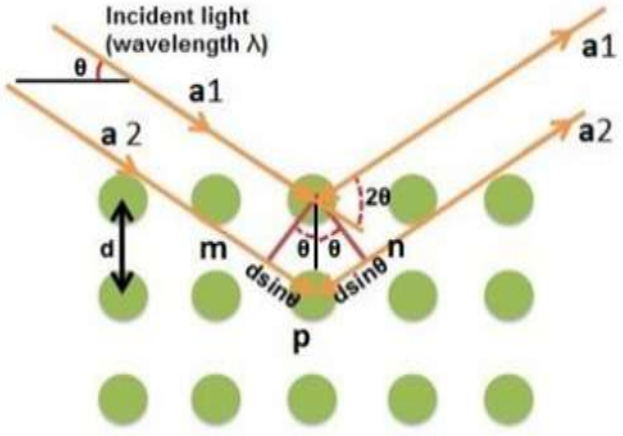


Fig-3: BRAGG'S Law

The conditioned can be satisfied if the distance mpn equals a multiple of complete wavelength (λ, 2λ, 3λ, 4λ or nλ)
The distance mp and pn equals to d sinθ

$$mpn = 2d\sin\theta$$

Where n=1, 2, 3…
λ=wavelength in A0
d=distance between the two planes of atoms (or) inter planar distance A0
θ= angle of incidence (or) angle of reflection of X-Ray beam
θ=sin-1(nλ/2d)

2.1 Mechanical Properties of Quasicrystals Investigated by SEM

Indentation and Scanning Probe Microscopes Hardness, elastic modulus and crack propagation were studied using micro- and Nano indentation, atomic force microscopy and scanning acoustic microscopy. We present measurements performed on icosahedral Al Pd Mn (three-dimensional quasicrystal) and decagonal Al Cu Co Si (two-dimensional quasicrystal) from room temperature to 550ÄC. Synthesis And Characterization of Al-Cu-Mn Quasicrystal Additionally, Y Mg Zn was impressed at room temperature. The surface of icosahedral specimens became fractured into segments exhibiting steps in height along shear cracks. Quantity of piling up as well as number and extension of cracks are

smaller for the two dimensional quasicrystalline material, which also displays a hardness anisotropy between different surface orientations. Quantitative hardness measurements revealed a strong indentation size effect exhibiting a hardness increase with decreasing load at room temperature, and the inverse behavior for higher temperatures.

3. RESULTS AND DISCUSSION

Measurement Conditions:

Sample Identification AlCuMn 75Hours

Raw Data Origin BRUKER-binary V2 (.RAW)

Scan Axis Gonio

Start Position [°2Th.] 5.0000

End Position [°2Th.] 100.0000

Step Size [°2Th.] 0.0190

Scan Step Time [s] 65.3153

Scan Type Continuous

Offset [°2Th.] 0.0000

Divergence Slit Type Fixed

Divergence Slit Size [°] 0.6000

Specimen Length [mm] 10.00

Receiving Slit Size [mm] 0.1000

Measurement Temperature [°C] 25.00

Anode Material Cu

K-Alpha1 [Å] 1.54060

K-Alpha2 [Å] 1.54443

K-Beta [Å] 1.39225

K-A2 / K-A1 Ratio 0.50000

Generator Settings 30 mA, 40 kV

Diffractometer Number 0

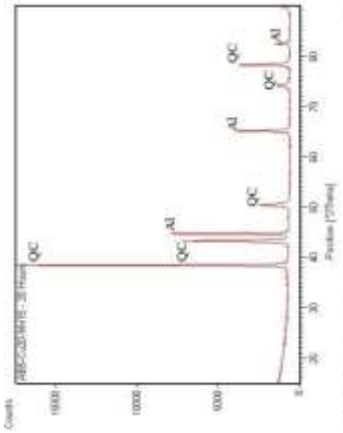
Goniometer Radius [mm] 217.50

Dist. Focus-Diverge. Slit [mm] 91.00

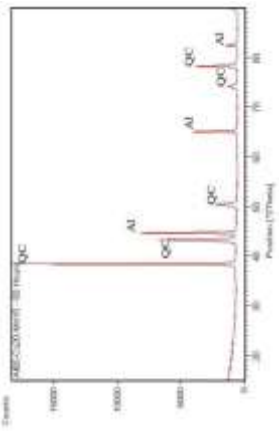
Incident Beam Monochromatic No

Table-1: Peak list

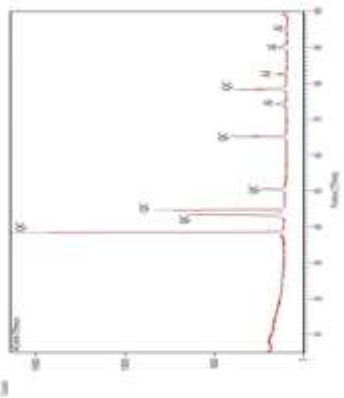
Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]
38.4490	15132.18	0.1140	2.33941	100.00
38.5683	8555.73	0.0684	2.33824	56.54
43.2990	5369.05	0.1596	2.08794	35.48
44.7013	7231.60	0.1140	2.02564	47.79
44.8338	4009.08	0.0684	2.02498	26.49
47.8289	132.52	0.3648	1.90023	0.88
50.4271	1401.91	0.2508	1.80824	9.26
65.0868	3265.48	0.1140	1.43195	21.58
65.2670	1845.11	0.1140	1.43198	12.19
74.1571	572.52	0.3192	1.27764	3.78
78.2203	2854.01	0.1140	1.22112	18.86
78.4533	1654.43	0.1140	1.22110	10.93
82.4370	771.07	0.1140	1.16901	5.10
82.6940	435.45	0.0684	1.16893	2.88
89.8992	431.85	0.3648	1.09033	2.85
95.1378	166.30	0.6384	1.04364	1.10
99.0727	328.38	0.2052	1.01246	2.17



Graph: 1



Graph:2



Graph: 3

Graph: X-Ray Driffraction Graphs Al_{65} Cu_{20} Mn_{15} (1)20, (2)50, (3)75 hours of milling.

Calculation by BRAGGS'S Law for 20 hours of milling

Table-2: 20 hours D-spacing

By BRAGG'S LAW nλ=2dsinΘ where n=1,2,3… λ=1.54	POS[2°Th]	D-spacing[A°]	
1ST PEAK : 2Θ=38° Θ=19°	38	2.3609	[QC]
1.54=2dsin(19°)	43.5	2.0779	[QC]
d=2.3609 A°	44.5	2.0335	[Al]
2nd peak: 2Θ=43.5° Θ=21.75°	50.5	1.8051	[QC]
1.54=2dsin(21.75°)	65	1.4330	[Al]
d=2.0779 A°	74	1.279	[QC]
3rd peak: 2Θ=44.5° Θ=22.25°	78	1.2235	[QC]
1.54=2dsin 22.5	82.5	1.1678	[Al]
d=2.0335 A°			

Calculation by BRAGGS'S Law for 50 hours of milling

Table-3: 50 hours D-spacing

By BRAGG'S LAW nλ=2dsinΘ where n=1,2,3… λ=1.54	POS[2°Th]	D-spacing[A°]	
1ST PEAK : 2Θ=38° Θ=19°	38	2.3609	[QC]
1.54=2dsin(19°)	43.5	2.0779	[QC]
d=2.3609 A°	44.5	2.0335	[Al]
2nd peak: 2Θ=43.5° Θ=21.75°	50.5	1.8051	[QC]
1.54=2dsin(21.75°)	65	1.4330	[Al]
d=2.0779 A°	74	1.279	[QC]
3rd peak: 2Θ=44.5° Θ=22.25°	78	1.2235	[QC]
1.54=2dsin 22.5	82.5	1.1678	[Al]
d=2.0335 A°			

Calculation by BRAGGS'S Law for 75 hours of milling

Table-3: 75 hours D-spacing

By BRAGG'S LAW nλ=2dsinΘ where n=1,2,3… λ=1.54	POS[2°Th]	D-spadng[A°]	
1ST PEAK : 2Θ=38.5° Θ=19.25°	38.5	2.335	[QC]
1.54=2dsin(19.25°)	43.5	2.0779	[QC]
d=2.335 A°	44.75	2.0227	[QC]
2nd peak: 2Θ=43.5° Θ=21.75°	50.5	1.8051	[QC]
1.54=2dsin(21.75°)	65	1.4331	[QC]
d=2.0779 A°	74	1.2794	[Al]
3rd peak: 2Θ=44.75° Θ=22.375°	78	1.2235	[QC]
1.54=2dsin (22.375)	82.5	1.1678	[Al]
d=2.0227 A°	90	1.0883	[Al]
	95	1.0443	[Al]

SEM results before milling Al Cu and Mn material grain structure

Fig-4: (a) Aluminum (b) Copper (c) Manganese

SEM results After 125 hours milling Al Cu and Mn material grain structure

Fig-5: after 125 hours milling Grain structure of Al Cu Mn

4. CONCLUSION

Al_{65} Cu_{20} Mn_{15} powder with a particle size smaller than 150 µm was milled for 125hours in planetary ball milling. After every 25 hours of milling the Al_{65} Cu_{20} Mn_{15} powder is subjected to characterization process for evaluation in X-ray diffraction and Scanning electron microscopy. Then the resulted graphs were indexed by calculating d-spacing value by using rag's law and these values were compared with standard values. As both the values are approximately similar it indicates Quasicrystals were successfully here. Conclusion content comes here synthesized.

ACKNOWLEDGEMENT

I would like to express my gratitude to Dr K T Kashyap and IISC Bangalore applied Materials staff. I believe I would not have been able to successfully complete this project. His subtle way of providing guidance and giving directions helped me to clarify my objectives and thus focus on achieving them. I would like to express my heartfelt gratefulness to him.

REFERENCES

[1] J. Eckert and L. Schultz "Formation of quasicrystals by mechanical alloying" Siemens AG, Research Laboratories, D-8520 Erlangen, Federal Republic a/Germany Received 24 February 1989; accepted for publication 3 May 1989

[2] B. Wolf*and P. PauÑer "Mechanical Properties of Quasicrystals Investigated by Indentation and Scanning Probe Microscopes" 27, 592È599 (1999)

[3] Xian-Fang LiandTian-You Fan "A Straight Dislocation in One-Dimensional Hexagonal Quasicrystals" phys. stat. sol. (b) 212, 19 (1999)

[4] Renhui Wang Chengzheng Hu(a), andJianlin Lei(b) "Theory of Diffuse Scattering of Quasicrystals Due to Fluctuations of Thermalised Phonons and Phasons" phys. stat. sol. (b)225, No. 1, 21–34 (2001)

[5] W. A. Wooster, Diffuse X-Ray Reflections from Crystals, Clarendon, and Oxford 1962.

[6] A. Guinier, X-Ray Diffraction, W. H. Freeman and Company, San Francisco and London 1963.

[7] M. Tanaka, M. Terauchi, K. Hiraga, and M. Hirabayashi, Ultramicroscopy 17, 279 (1985).

[8] P. A. Bancel, P. A. Heiney, P. W. Stephens, A. E. Goldman, and P. M. Horn, Phys. Rev. Lett

[9] Thierry Klein, Claire Berger, Didier Ma you,* Franqoise Cyrot-Lackmann "Electronic Properties of Quasicrystals" 1985