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Microstructure and Bending behaviour of Thixocast 2014 Alloy : Effect of processing temperature

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Abstract

In the present work, microstructure and mechanical properties of thixocast 2014 alloy were correlated and compared with gravity cast 2014 alloy having the same composition. Thixocasting of samples was done at 600°C, 615°C & 630°C within a cylindrical die. Microstructures and mechanical properties were observed, correlated and compared with those of gravity cast samples. The tensile strength, yield strength, hardness and percentage elongation of the thixocast samples found to be higher than those of gravity cast samples. Improved mechanical properties of thixocast samples are due to non-dendritic globular structure and morphology of silicon particles.

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Introduction

Semi-Solid metal processing, invented more than 30 years ago at Massachusetts Institute of Technology [1], a metal forming process that fills partially-solidified metal with globular structure in a mould, instead of casting with liquid metal. In 1972, the first paper was published on the topic of thixocasting. The experiment was performed on the Tin-lead alloy in which alloy is heated between the range of solidus and liquidus line and casted in a die by the application of external pressure. It was found that thixocast material has higher mechanical properties as compared to conventional casting. Therefore, properties of the material by thixocasting became a point of attention for the researcher. In semi-solid metal processing technique lower amount of heat is required due to the partial liquid position of the metal, causing higher viscosity of the material [2]. Therefore an external pressure is applied on the material during casting to minimize the micropores present in the final product. Semi solid casting has number of advantages over the conventional liquid metallurgy processing of the material. By the use of this technique, thin walled complex shape product of high dimensional accuracy with improved mechanical properties can be obtained.

In the thixocasting process, continuous stirring is done to find the globular shape of the sold phase material in the liquid phase. The presence of globular shaped particles in the liquid provides better flowability of the material during thixocasting which reduces the pores present in thixocast sample. So, macro-segregation, porosity and forming forces during shaping process can be minimized. The advantages of thixocasting are higher product quality, lower forming temperature and higher production rate with net shape and complex geometry metal components [4, 5]. As a semi solid slurry an alloy has a much higher viscosity than when fully liquid, thereby retaining laminar flow and filling the die more evenly, facilitating the near-net shape forming with a single step process [6]. It was has been proved many times that when ratio of liquid and solid phase present in the metal mixture is 1:1 then performance of the product enhance [7]. It can be said that Thixo-forming is the combination casting and powder metallurgy route of the processing which enabling the complex shaped product [8], [9]. For a successfully thixoformed, Al alloy must be exhibit a non-dendritic microstructure, more precisely, one which is formed by a equiaxed primary phase (Al-α) well dispersed into a eutectic “liquid matrix”. Equiaxed primary phase favours the rheological behaviour which provides better flow property of the alloy in the cavity of the mould [10]. The forming process in a semi-solid state is, however, so useful from the point of view of the shape variations of the product or resulting microstructures, that many innovative variations of this unconventional technology can be expected in the future [11]. Semi solid metal processing or thixo forming is found more suitable in case of Al alloys. As Al is a light metal and lower melting point, it is more effective for the purpose of weight reduction, especially in automobile sector with ease in product manufacturing. Semi Solid Processing of aluminium alloys has already been implemented in Industries. Some existing limitations are associated with the design of dies so that the possible defects can be eliminated [12]. It is not practical to cast thick parts in conventional die casting, since so much heat needs to be extracted that the die life is significantly shortened and productivity is lower. Semi Solid Metal processing, thus, allows die casting to be used to produce a wider range of products. Besides high-pressure die casting applications, recently gravity casting of Semi Solid Metal with low solid fractions into a mould has been demonstrated [13]. In high-pressure die casting applications, parts can be produced with higher quality because less turbulent flow is obtained during the mould filling, thereby producing parts with minimal air entrapment and oxide inclusions. The higher quality consequently gives the parts with higher mechanical properties and allows them to be heat-treated, machined, anodized, and welded. In addition to a higher part quality, the production cost of parts produced by Semi Solid Metal processing is lower than of those, produced by conventional liquid pressure die casting

[14]. Semi Solid Metal slurry cast into a die requires significantly less heat to flow into the die before the part can be removed. As a result, the die operates at a lower temperature and the die life increases. In addition, since less heat needs to leave the part, the cycle time can be significantly shorter resulting in an increase of the productivity [15]. These factors result in a significant reduction in operating cost when compared with conventional die casting. One of the goals of the transport industry is weight reduction; indeed, it has been shown that replacement of steel by Al alloys can lead to a 20–30% saving which translates into fuel economy and lower tail pipe emissions. These two final objectives can of-course be met by improvements on combustion efficiency, but the use of light materials is expected to be much more effective. Thixocasting may be reasonable for thick-walled parts with rather simple geometries, where it can provide sound castings with low porosity and good ductility. Such parts cannot be produced by HPDC and are supposed to be weldable, pressure tight and heat treatable. Due to quite coarse grains in the thixocast parts, only medium yield strength values are achieved [16] In present time, efforts for the development and implementation of thixocasting done on entire world because these offers many advantages as compared to the conventional processing methods (casting in liquid state and forging, die-forging, stamping in solid state), advantages that come out of the behaviour and characteristics of the materials in semisolid state. So, due to the heat content, lower than that of the liquid metal, high processing speeds can be applied, the wear of the deformation tools being lower. Thixocasting represents a paradigm change in casting. The flow behaviour enables the use of hot runners making casting more competitive with the plastics industry [17].

Experimental Procedure

Synthesis of 2014 Alloy

2014 alloy is melted in the electric resistance furnace at temperature range of 700-720°C. Coveral 11 is used as cover flux and dry Nitrogen gas as degasser. Then, during the stirring operation (approx. 500-600 rpm for 3-4 minutes), Al – TiB_2 master alloy was added in alloy melt prior to pouring into the die for casting to achieve relatively globular dendritic structure and grain refinement of matrix material. The liquid alloy has been solidified in preheated cast iron molds. The chemical composition of 2014 alloy is given in Table-1.

Table 1 - Chemical Composition (weight %) of 2014 Alloy

Cu	Mg	Mn	Fe	Zn	Ti	Cr	Si	Al
4.5	0.5	0.4	0.5	0.1	0.15	0.05	0.8	Remainder

Fig. 1 2014 alloy Finger Castings

Thixocasting

The billets of alloy samples (75×210 mm) were used as stock. These feed stock were heated within cylindrical die (115×40 mm) at 600°C, 615°C & 630°C to achieve various amount of liquid phases. Finally, these billets were pressurised within the closed cylindrical die by using a 400 ton pressure die casting machine. Samples were prepared in the cylindrical shape of diameter 15 to 25 mm. The microstructures of the samples were observed and the phases formed were identified. Optical Microscope (Model : RMD-MPD-EQP-1 Leitz, METALLOPLAN, Germany) and Scanning Electron Microscope (Make : FEI) were used. The phases formed were identified by X- Ray diffraction (Model : Bruker-D8 with Cuα radiation). The mechanical properties were measured by UTM (Instron make, Model 8801).

Fig. 2 Die Used for Thixocasting

Microstructure Characterization The alloy samples were cut into cube samples of 25mm size and used for microstructure characterization. The samples impregnated with mounting material and then polished & etched using standard metallographic techniques. The polished samples etched in Keller's reagent (2 ml HF +3 ml HCL + 5 ml NO3+ 190 ml water). The microstructures were observed under an optical microscope (Model : RMD-MPD-EQP-1 Leitz, METALLOPLAN, Germany) and Scanning Electron Microscope (Make : FEI). Samples were gold sputtered prior to SEM examination. The grain size determination has been done by Intercept Method (as per ASTM E112-13). Volume fraction determination was carried out by Point Counting Method (as per ASTM E562-11). Fracture surface study has also been done by Scanning Electron Microscope (Make : FEI) for analysing mode of failures of the specimen during tensile loading. The types and causes of fractures in the material under study were interpreted on the basis of fractography.

XRD Analysis

The physical and chemical changes in phase constituents in the alloy were examined by X-ray diffraction (Model : Bruker-D8 with Cuα radiation) at a scan rate of 0.02°per Second. The 2Θ angle was varied from 10° to 90° and the scanning rate used was 1.2° per minute. D-values obtained from XRD patterns were compared with the characteristic d-spacing of all possible values from JCPDS cards to obtain the various X-ray peaks.

Mechanical Properties

Hardness Test

Vickers's Hardness Tester / Micro Hardness Tester (Model: LEICA VMHT 30A) has been used to measure hardness of the gravity cast and thixocast samples, at 1 kg loading. For microhardness test the specimens were sectioned small enough so that it could fit into the tester. Also, the specimen's surface was smoothed enough to allow a regular indentation shape and to ensure that it could be held perpendicular to the indenter. For each sample, hardness was measured at twenty five different locations and the average of these values is taken for analysis of results.

3-Point Bending Test

3 Point Bending or Flexural test is a method to determine the flexural strength of materials. This test was carried out for alloy and composite, gravity cast and thixocast samples, on UTM (Instron make, Model 8801) with 3 point bend fixture with 70mm span length. For each category, samples were tested and the average value is taken for analysis of results.

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Results & Discussions

Thixocast Alloy Before and After machining

The feed stock was thixocast into simple cylindrical billets. The feedstock had the dimension of 40mm×115 mm. These feed stock were again melted in semi-solid regions and cast as per the Al-Si phase diagram. The extent of liquid and solid varies with the variations of temperature of casting. As the casting temperature increases, the volume fraction of liquid phase in the feed stock increases. Hence during casting , the microstructure as well as mechanical properties changes with casting temperature. The volume of feed stock was intentionally made slightly higher as compared to the volume of die cavity. The excess material in feed stock get splashed out of the die cavity after casting (Fig. 3 a). The material flow during casting also visible from the lateral surface of the thixocast billet. When these billets are machined around 1mm the surface does not show any cracks (Fig. 3 b). The density of these machined thixocast samples was also measured. It was noted that the density of thixocast samples is about 2.85 gm/cc and that of gravity cast samples was 2.80 gm/cc. This signifies that thixocast alloy samples are more dense than the gravity cast ones. Thixocast samples will have less defects like blow holes and porosity. The machined billet of thixocast samples (at different temperatures) (Fig. 3 c) showed that the thixocast parts are very sound at every temperature of casting.

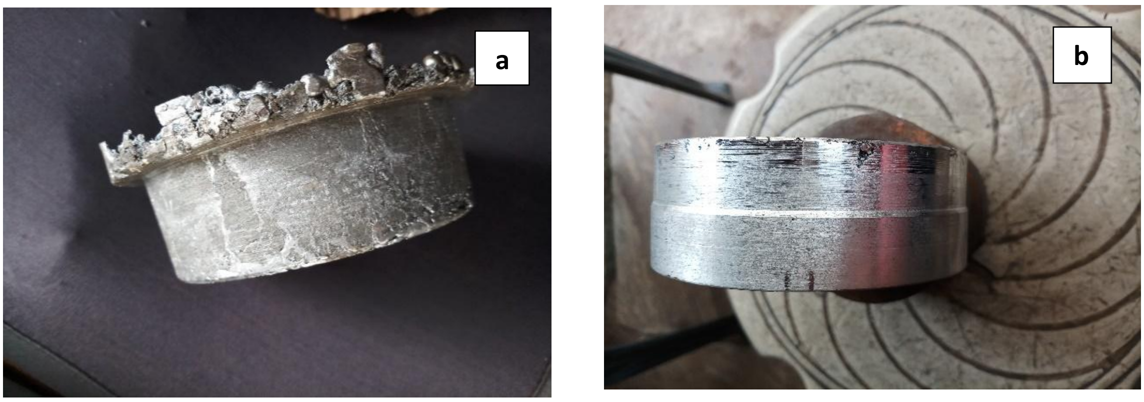

Fig. 3 Thixocast Alloy : (a) Before Machining (b) After Machining

Fig. 3 (c) Thixocast Alloy After Machining (at diff. processing temperatures)

Microstructures : Gravity Cast and Thixocast 2014 Alloy

At high processing temperatures, melting of $CuAl_2$ eutectic phase present in α-Al boundaries takes place. As a result, we get solid with dendritic morphology surrounded by liquid. At this stage thermodynamic condition of minimum solid-liquid surface energy is obtained by converting the dendritic morphology of the solid phase to spherical morphology. The coarsening and coalescence result in the globular structure required in the thixocast material.

In 2014 alloy, the structure in the gravity cast samples consists of α-Al with a very homogeneous distribution of Cu in solution with dendritic morphology and eutectic present between dendritic arms and grain boundaries. Eutectic is lamellar mixture of α phase and micro constituent $CuAl_2$.

In gravity cast samples a typical dendritic shape of the α-Al phase was observed, whereas in thixocast samples a non-dendritic (spherical) primary α-Al phase was observed. The samples thixocast at 600°C shows a very small level of porosity. Samples thixocast at 615°c showed little more globular α-Al particles. At 615°C the primary α-Al phase was more continuous as compared to 630°C processing. The eutectic Si phase and α-Al phase in thixocast samples changes with the temperature of casting. The size of α-Al phase here considered as dendrite size or grain size. The sphericity of the dendrite is noted to be higher in case of thixocast samples as compared to gravity cast one. The concentration of α-Al phase decreases with increasing thixocasting temperature. This is quite obvious from the Al – Si phase diagram. It is noted from this observation that casting at higher temperature causes more fluidity and thus casting become more easy. But at the same time, after casting , there is a possibility of coarser dendrite size. This will also cause difficulty in casting. As a result, there would be chance of elongation of α-dendrites. As a result, the aspect ratio of α-Al phase (grain) increases marginally. But, in all the cases for thixocasting, the aspect ratio varies in the range of 0.71 to 1.05, which indicates almost spherical shape of the secondary dendrites in the matrix (Table-2). Under pressure, cooling rate may be more. But, when the sample is heated at higher temperature, there is a possibility of growth of α- dendrites and its merger. But, the size of Si in eutectic phase reduces and become more fibrous type when thixocast at higher temperatures. This causes eutectic phase to be stronger when thixocast at higher temperatures. The overall effect of these microstructural characteristics causes an improvement in strength and hardness of the thixocast samples as compared to that of gravity cast ones (Table-2). Excellent mechanical properties of thixocast specimens are not only due to nondendritic microstructure but also due to the small size of the primary α- Al, which specially results in enhanced elongation to fracture. When the shape factor increases (more rounded primary globules), tensile and elongation properties also increases. However, because of the microstructural variation, there is a possibility of optimum temperature of thixocasting for getting maximum strength and hardness.

Table 2 – 2014 Alloy

Type of Processing	Grain Size (µm)	Aspect Ratio	Hardness HV	Volume Fraction	
				Eutectic	α – Al
Gravity Cast	44	1.40	86	68	32
Thixocast at 600°C	143	1.05	112	75	25
Thixocast at 615°C	110	1.04	108	76	24
Thixocast at 630°C	115	1.23	107	81	19

Hardness

The hardness of alloy increased after thixocasting. This is due to strain hardening effect during deformation caused by thixocasting. Low porosity level and increased dislocation density is obtained with the application of pressure during solidification, resulting in the improved tensile properties and increased primary α phase hardness.

Tensile Properties

The results obtained show that the tensile strength, yield strength and elongation to fracture for the thixocast alloy was greater than those of gravity cast samples. In gravity cast samples, typical dendritic shape of primary α phase was observed. The improvement in mechanical properties is due to the non-dendritic structure produced and morphological aspects of the

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silicon phase. The effect of applied pressure in thixocast samples is more significant as compared to gravity cast samples. As a result of applied pressure the tensile property is improved due to low porosity level and increased dislocation density. Increase in applied pressure results in reduction of grain size which promotes the improvement in mechanical properties.

Table 3 - Three Point Bending Behaviour of 2014 Alloy

Type of Processing	Flexure Strength (MPa)	Max^{m} flexure extension (mm)	Flexure Stress at Max^{m} flexure extension (MPa)	Modulus (GPa)
Gravity Cast	288	1.30	40	66
Thixocast at 600°C	385	1.50	108	97
Thixocast at 615°C	372	1.70	95	96
Thixocast at 630°C	368	2.08	85	85

Fractography

In case of 2014 Al-alloy, the inter-dendritic phase is very low. These are very brittle in case of bending, mainly shear type deformation is taking place. As a result, the eutectic phase is easily sheared and cracks are generated through these eutectic phase (Fig 4(a)).This cause delamination of dendrites during three point bend testing, which causes dimple type structures, even though the material behaves like brittle material. At higher magnifications, these observations become more clear (Fig 4(b)). Due to thixocasting, the defects in casting are reduced, eutectic get refined and dendrites become finer and cellular. As a result, the eutectic become more ductile and showed mixed mode fractures. Fine serration are noted along eutectic (Fig 4(c)). But the dendrites get decoheted (Fig 4(d)) as a whole. Similar trend of fracture surface is noted, when the alloy is thixocast at 615°C (Fig 4(e)). At higher thixocasting temperature (630°C), in addition to decohesion micro-cracking along the

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boundary of decoheted dendrites are noted (Fig 4(f)), which become more clear at higher magnification (Fig 4(g)). This causes lower flexural strength.

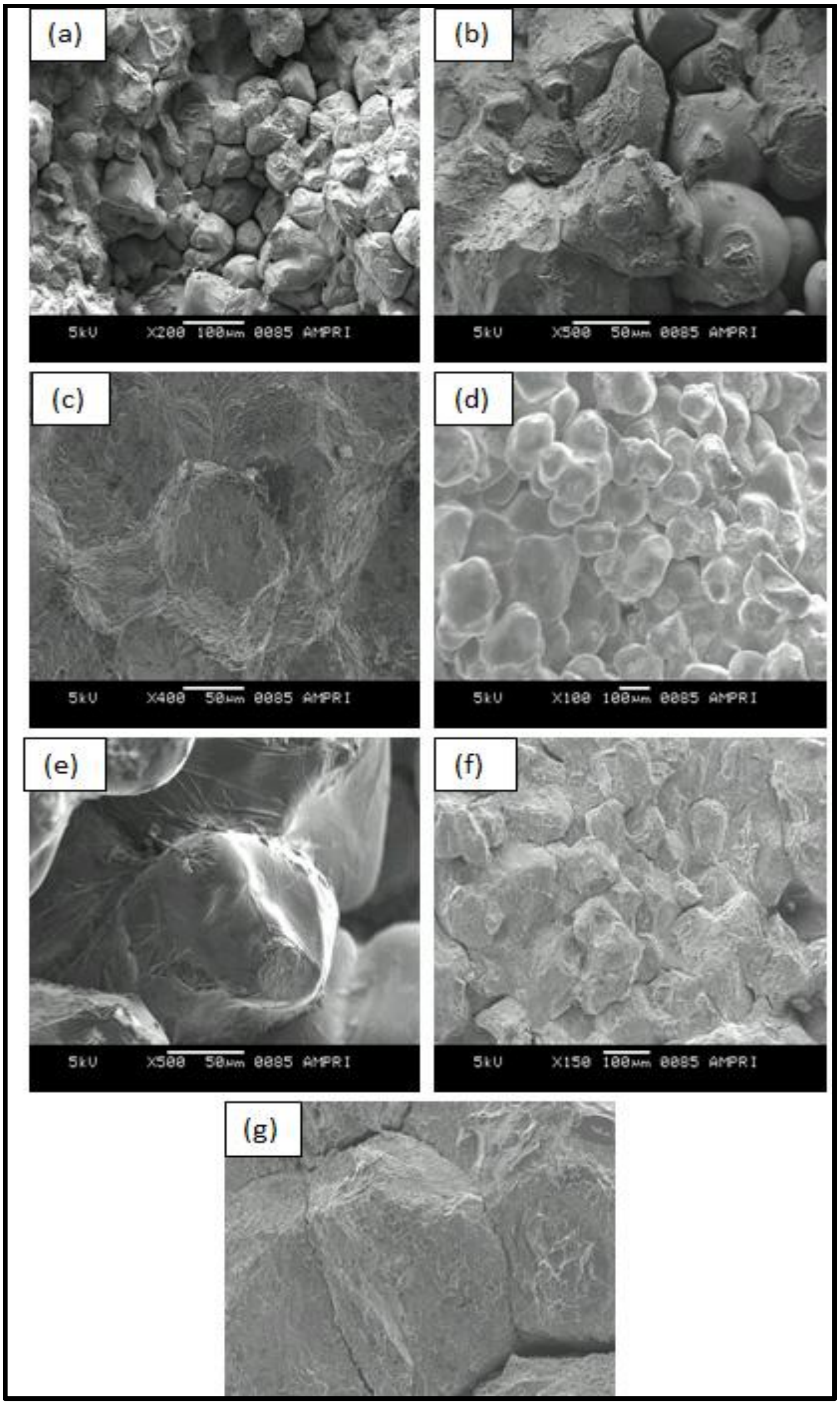

Fig. 4. 2014 Alloy : Bending Fractographs - (a) & (b) Gravity Cast, (c)Thixocast at 600°C, (d) & (e) Thixocast at 615°C and (f) & (g) Thixocast at 630°C

Conclusion

Thixocast 2014 alloy samples were found with significant improvement in mechanical properties as compared to the gravity cast samples due to fine and globular microstructure. The improvement in mechanical properties is due to the non-dendritic structure produced and morphological aspects of the eutectic phase. Microstructural changes and morphological aspects of eutectic phase causes the difference in the tensile fracture paths. The possibilities of fracture increases with long and elongated silicon particles as compared to spherical α-Al dendrites. The presence of porosity act as fracture initiation points in gravity cast samples. It result in very low level of ductility and strength. As compared with gravity cast samples, thixocast samples were found with low porosity level and higher dislocation density. The size of dendrite and volume fraction of eutectic phases changes with thixocasting temperatures which also causes changes in properties and hardness with thixocasting temperature. In samples thixocast at 600°C, a very small level of porosity with finer and more globular dendrites, was observed which is responsible for better properties. Thixocast samples of low processing temperatures shows improved properties due to the low aspect ratio of $CuAl_2$ particles along with low porosity level. Coarsened $CuAl_2$ particles provides sources for stress concentration in thixocast samples processed at 615°C & 630°C due to higher aspect ratio of $CuAl_2$ particles as compared to thixocast samples processed at 600°c. Thixocasting process can be very beneficial in improving mechanical properties of 2014 alloy as compared with gravity casting. Thixocasting at lower temperature is expected to give even better microstructure and mechanical properties. Irrespective of loading conditions (tensile, compressive or bending) thixocast samples provide higher strength and hardness. Amongst the thixocast samples, the alloy samples thixocast at lower temperature exhibited higher mechanical properties and finer microstructure. In tensile and bending fractures, dendrites fracture due to shearing, that caused ridges, which confirms the brittleness.

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