

Microstructure and Compressive 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 compressive strength, hardness and strain 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.

Introduction

Semi-Solid metal processing, invented more than 30 years ago at Massachusetts Institute of Technology. It is a metal forming process that fills partially-solidified metal with globular structure in a mould, instead of casting with liquid metal [\[1\]](#page-6-0). The first published experiments with the utilization of thixo-properties of metals were carried out in 1972. These experiments were done with tin-lead alloys at a temperature interval between solidus and liquidus, thus in a semi-solid state. These properties almost immediately became the centre of attention of many research groups, which investigated the hidden potential of this method. The characteristics of Semi-Solid metal processing are - lower heat content than liquid metal, partially-solidified metal at the time of mould filling, higher viscosity than liquid metals, flow stress lower than for solid metals [\[2\]](#page-6-1). These characteristics offer several potential benefits for various applications. Semi-solid metal processing enables the manufacturing of components with complex shapes, with thin walls, with good mechanical properties and with a high dimensional tolerance and accuracy. The thixocasting process uses stirring of the melt during the solidification of a continuous cast bar to obtain the globular microstructure. These bars are then cut to the required pieces and reheated to the hot-working temperature. Semisolid metal processing (SSM) is a new technology that offers several advantages over liquid processing and solid processing. This process utilizes semisolid behaviour as well as reduces macro-segregation, porosity and forming forces during shaping process. A lot of research work has been carried out by various researchers in order to exploit the potential of this process to produce different products especially for automotive industry [\[3\]](#page-6-2). It has been shown that semi solid metal forming processes have several advantages over other conventional forming processes. These include higher product quality, lower forming temperature and higher production rate [\[3\]](#page-6-2). Semi-solid metal processing is a modern metal forming technology offering net-shape metal components of complex geometry in a one-step operation [\[4\]](#page-6-3). 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 [\[5\]](#page-6-4). It was demonstrated and confirmed many times that the most stable rheocast flow occurs in around 50% solid slurry [\[6\]](#page-6-5). Thixoforming combines the advantages of casting and forming thus enabling the production of components with very complicated shaped designs [\[3,](#page-6-2) [7\]](#page-6-6). Although a number of metallic materials are being considered, presently the aluminium alloys appear to be the most suitable choice for the process. However, to be successfully thixoformed, these materials must 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". This microstructure exhibits a favourable rheological behaviour which gives good flow characteristics of the alloy into the mould cavity [\[8\]](#page-6-7). The aim was particularly to obtain a globular microstructure. Since the first research concerning the forming of steels in a semi--solid state, which were published at the beginning of 1980s, a lot of interesting results have been published. 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 [\[9\]](#page-6-8). Semi solid metal processing or thixo forming is found more suitable in case of Al alloys. As Al is a light metal, it is more effective for the purpose of weight reduction, especially in automobile sector. 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 [\[10\]](#page-6-9). 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 [\[11\]](#page-6-10). 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 [\[12\]](#page-6-11). 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 [\[13\]](#page-6-12). 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 [\[14\]](#page-6-13). Today, 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 [\[4\]](#page-6-3).

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), $AI - TiB₂$ 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.

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.

Mechanical Properties

Compression Test

The compression test of as cast and thixocast samples, were carried out at a strain rate of 0.01per second on cylindrical samples of 10mm diameter and 15mm length. During the test the surfaces between the anvil and specimen were lubricated with $MoS₂$. The stress and strain were recorded in the system interfaced with UTM (Instron make, Model 8801). For each category, samples were tested and the average value of tests is taken for analysis of results.

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.

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 (c). Thixocast Alloy After Machining (at diff. processing temperatures) Microstructures: Gravity Cast and Thixocast 2014 Alloy

At high processing temperatures, melting of CuAl₂ 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 (Fig 5 (a)). 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-Al phase and micro constituent of CuAl₂. In gravity cast samples a typical dendritic shape of the α -Al phase was observed (Fig 5(b)), whereas in thixocast samples a non-dendritic (spherical) primary α-Al phase was observed (Fig 5(c)). 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 (Fig 5(d)). At 615°C the primary α-Al phase was more continuous as compared to that at 630°C processing (Fig 5(e)). The CuAl2 eutectic 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 (Table 2). This is evident from the Al – Cu 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 0.98 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 (Fig 5(f)). 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 aspect ratio increases (more elliptical), tensile and elongation properties decreases. However, because of the microstructural variation, there is a possibility of optimum temperature of thixocasting for getting maximum strength and hardness.

4 (a) 2014 Gravity Cast 4 (b) 2014 Gravity Cast

4 (e) 2014 Thixo 615°C 4 (f) 2014 Thixo 630°C

4 (c) 2014 Thixo 600°C 4 (d) 2014 Thixo 615°C

Hardness & Compressive Properties

The hardness of alloy increased after thixocasting. This is due to strain hardening effect during deformation caused by thixocasting and also due to microstructural refinement and modification. 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. At higher processing temperature, size of α-phase is more, strain hardening and dislocation density will be less. As a result, the hardness and strength decreases with increase in processing temperature. The results obtained show that the compressive strength and strain at failure 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 CuAl₂ eutectic 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 compressive property is improved due to low porosity level and increased dislocation density. Increase in applied pressure results in reduction of globular dendrite size which promotes the improvement in mechanical properties. The compressive stress-strain diagram is shown in Fig 5. This clearly demonstrate that the strain is almost invariant to the casting conditions. But compressive strength of thixocast samples are higher than that of gravity cast one. Again, the strength noted to be decreased with increase in thixocasting temperature. The trend is same as that of hardness. At higher thixocasting temperature the sphericity of globular dendrite may be almost same, but the volume fraction of dendrite decreases and eutectic CuAl₂ increases. This is quite obvious from the phase diagram of Al-Cu alloy. At higher temperature the dendrites are found to be coarser in size and the CuAl₂ eutectic also become coarser. This lead to reduction in strength of thixocast samples with higher casting temperature.

Fig. 5. Compressive Stress-Strain diagram

Conclusion

Thixocast 2014 alloy samples were found with significant improvement in compressive 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 fracture paths. The possibilities of failure 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 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 compressive properties and hardness. In samples thixocast at 600°C, a very small level of porosity was observed which is responsible for better compressive properties. Thixocast samples of low processing temperatures shows improved tensile properties due to the low aspect ratio of CuAl² particles along with low porosity level. Coarsened CuAl₂ particles provides sources for stress concentration in thixocast samples processed at 615°C & 630°C due to higher aspect ratio of CuAl² 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.

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