Effect of Semi-Solid Processing on Microstructure and Tribological Characteristics of Al-10Cu alloy

Thesis submitted in partial fulfillment for the Award of Degree of

Doctor of Philosophy

By

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It is certified that the work contained in the thesis titled ***"Effect of Semi-solid Processing on Microstructure and Tribological Characteristics of Al-10Cu alloy"*** by ***" Sankara Rao L."*** has been carried out under our supervision and this work has not been submitted elsewhere for a degree.

It is further certified that the student has fulfilled all the requirements of Comprehensive, Candidacy and SOTA.

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I, *" Sankara Rao L."*, certify that the work embodied in this thesis is my own bona fide work and carried out by me under the supervision of ***"Prof. S. N. Ojha and Prof. A. K. Jha"***from ***"July 2012"***to ***"Jan 2018"***, at the ***"Department of Metallurgical Engineering"***, Indian Institute of Technology (BHU), Varanasi. The matter embodied in this thesis has not been submitted for the award of any other degree/diploma. I declare that I have faithfully acknowledged and given credits to the research workers wherever their works have been cited in my work in this thesis. I further declare that I have not willfully copied any other's work, paragraphs, text, data, results, *etc.*, reported in journals, books, magazines, reports dissertations, theses, *etc.*, or available at websites and have not included them in this thesis and have not cited as my own work.

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Sankara Rao L.

Dedicated to

My Family

and

Supervisors

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PREFACE

Aluminium alloys find a wide range of applications in automotive and aerospace industries due to their high specific strength and ability to be cast into complex shape. However, a cast product is known to have coarse dendritic structure and macro segregation. Consequently, there is continuous endeavor to the development of new processing techniques for in-process modification of the microstructure. The semi-solid processing techniques fulfil this criterion and research efforts in this direction have led to development of a large number of processing techniques. In these techniques, the alloys are held in semi-solid region at different temperature and time. These are also subjected to the stirring to create a slurry and cast into a die for making desired shape of the component after solidification of the melt. Even though there are many variants of semi-solid processing techniques, a proper understanding of liquid distribution in the matrix phase and its solidification behaviour have not been systematically studied. These understanding will facilitate processing of even unconventional alloy with their effective control of the microstructure and better tribological behaviour. The present investigation has been carried out with this objective. A wide freezing range Al-10Cu alloy has been selected for processing employing different semi-solid processing techniques and critical evaluation of their tribological characteristics.

This thesis consists of five chapters. **Chapter-1** presents an overview of semi-solid processing techniques and their characteristics features. The microstructural features of the alloy and techniques employed for their characterization have been discussed. The morphological features of the primary phase and eutectics and mechanisms of their formation have been brought out.

Chapter 2 comprises the investigation of solidification of liquid phase entrained in its primary solid. A hypo-eutectic alloy based on Al-Cu-Fe system containing Fe and Si was thermal cycled between semi-solid region to low temperatures. The freezing characteristics of the liquid were recorded in inverse rate cooling curves. The continuous network of the liquid phase progressively changed into isolated droplets with their different size and size distribution. Such droplets revealed undercooling of the melt varying from 20 to 35°C below the eutectic temperature of the alloy. This behaviour of melt undercooling is discussed in light of independent nucleation events associated with freezing of droplets. Solidification structure of droplets revealed particulate eutectic phases in contrast to lamellar eutectic microstructure in the inter-dendritic region of the as cast alloy. The decomposition of supersaturated solid solution phase resulting from large undercooling of droplets were explained for this anomalous behavior of the eutectic. The hardness of the matrix as well as that of droplet solidified areas were examined and found 152 HV for the matrix and 267 HV for its droplets. The wear property of the alloy was studied in a range of load varying from 10-50 N at sliding velocity 1.0 m/s. The droplet distribution and their solidification structure resulted in an improvement in tribological characteristics of the alloy. This effect is attributed to the presence of ultrafine solidification structure of droplets arising from their large undercooling. This feature is evident in the microstructure of droplet regions. In addition, the solute elements are themselves get redistributed differently in droplets vis-a-vis that in the liquid present in the inter-dendritic region of the cast alloy. The EDAX analysis of droplet regions and that of the bulk liquid region provides support in this direction. This effect is correlated with features of wear surfaces generated on the matting surfaces.

Chapter-3 includes the investigation of Al-10Cu alloy containing small amount of Fe and Si which were produced by rheocasting process at different mechanical stirring speeds. The resultant microstructures and wear properties were compared with that of a conventional metal mould cast alloy. The average grain size was measured to be 55 μm for the 800 rpm and 30 μm for the alloy melt stirred at 1200 rpm samples respectively. The rheocast alloys have shown better mechanical (ultimate tensile strength and hardness) and wear properties as compared to metal mould cast alloy. Moreover rheocast alloy which was produced at 1200 rpm stirring speed exhibited enhanced wear and mechanical properties as compared to other rheocast alloys. The improved wear rate for this alloy was attributed to finer grain size and the nearly-spherical morphology of the primary α - phase. The metal mould cast alloy and rheocast alloy at 400 rpm have shown adhesive wear condition whereas, other rheocast alloys have displayed microcutting mechanism of wear. The noticeable decrease in the average roughness for 3 m/s sliding velocity was observed as compared to a sliding velocity of 1 m/s. In addition, the average roughness value of the 1200 rpm stirred alloy is observed to be lower than that of the alloy produced at 800 rpm stirring speed.

Chapter-4 deals with the study of influence of synthesis methods, namely, the conventional metal mould casting (MMC) followed by strain induced melt activation (SIMA) processes, on the microstructure and tribological characteristics of Al-10Cu alloy. The alloy was prepared by melting the commercial purity aluminium and copper. The effect of holding time in semi-solid region on the microstructure of the alloy subjected to SIMA was studied. Microstructural characterization was performed using optical and scanning electron microscopy, while chemical segregation is investigated using energy dispersive spectroscopy. Hardness and strength of the resulting alloy are measured using macroindentation and tensile testing respectively. Near-spherical grains were achieved in the SIMA process with an average grain diameter varying from 45to 65 μm for holding times ranging from 30 to 55 minutes. The wear properties of stirred MMC alloy which was subjected to SIMA process are considerably better than those of either conventional MMCs or unstirred MMC subjected to SIMA process. The 50 % pre-deformation followed by intercritical annealing at 580 ° C and 30 minutes holding time are the optimum parameters for to obtain such a wear properties of Al-10Cu alloy. The improved wear and mechanical properties of the alloy are discussed in light of the microstructural features of SIMA processed alloy and the nature of the worn surfaces.

Chapter-5 presents the study of producing Al-10Cu alloy by vertical centrifugal casting at speeds ranging from 800-2850 rpm. The microstructural features, mechanical and wear properties have been investigated. The wall thickness of stirred centrifugal cast Al-10Cu alloy is uniform from top to bottom resulting in a sound casting. The variation in thiekness of casting from top to bottom is 25 mm for 800 rpm, 7 mm for 1320 rpm, 1 mm for 1980 and 10 mm for 2850 rpm. The microstructure of Al-10Cu alloy consists of equiaxed grain morphology of the primary α-phase with eutectic phases in the interdendritic regions. It has been observed that there is a variation in the grain size from the inner surface of the casting to its outer surface. The speed also has a strong influence on the grain size and subsequent mechanical properties of the alloy. The tensile strength and hardness of stirred centrifugal cast alloy were observed to be 181 MPa and 150 HV which are higher than that of the other alloys. The wear properties of the alloy have been evaluated at a constant sliding velocity of 1 m/s for a range of applied load and sliding distance. The optimum mould speed was found to be 1980 rpm as it produced fine microstructure in the casting resulting in optimum hardness, ultimate tensile strength and wear rate. The cumulative volume loss and consequent wear rate of stirred centrifugal cast alloy are invariably lower than that of the other alloys under study. The worn surface of stirred centrifugal cast alloy appears to be less damaged than that of the other centrifugal alloys. This is because of the reason that the driving force acting on the molten metal at optimum speed was sufficient enough to carry the molten metal to the inner surface of the mould and hold firmly there before it get solidified resulting in a uniform cylindrical casting. The variations in the wear behavior are attributed to the size and solidification morphology of the castings.

The conclusions of the present thesis work are summarised and have been presented in the section that follows. It is noteworthy to observe that amongst various semi-solid processes investigated in the present work, the SIMA process resulted in considerable refinement of the primary phase with change in its morphology to round shape. The constituent of the eutectic phases in the inter-dendritic region exhibited globular shape. The understanding of morphological changes have been explained on the nature of distribution of liquid explained on the nature of distribution of liquid either along grain inter-dendritic region or within the solid phase as isolated droplets. The detail study of the solidification behaviour of such liquids exhibit several interesting features and considerable change in tribological characteristics of the alloys. These features are discussed in detail.

Overall Conclusions

Thermal cycling of a hypoeutectic Al-10Cu alloy showed distribution of isolated droplets in the primary solid phase. Initial stages of thermal cycling resulted in liquid along the grain boundaries. However, in subsequent thermal cycling, the continuity of the grain boundary liquid was observed to break down to discontinuous liquid which formed isolated droplets. The solidification structure of continuous liquid exhibited regular eutectic in contrast to particulate eutectic microstructure of the isolated droplets. Distribution of droplets and their resultant solidification structure led to an improvement in the wear property of the alloy compared to the alloy processed by normal casting route.

In rheocasting the primary α - phase is changed from dendritic to nearly spheroidal morphology arsing from stirring action of the melt. The finer grain size of primary α - phase was observed at maximum rotational speed of 1200 rpm. This observation of increased grain refinement with higher stirring speeds can be rationalized by increased fragmentation and uniform distribution of solid particles in the slurry at higher stirring speeds resulting into pronounced decrease in grain size. The cumulative volume loss of all alloys increased with increase in load irrespective of the processing method. The coefficient of friction values for rheocast samples were observed to be less than the metal mould cast alloy. The wear rate of metal mould cast alloy rapidly increased beyond 3 m/s sliding velocity. For rheocast alloys, 4 m/s is critical sliding velocity and beyond this wear rate is increased drastically.

In the SIMA process a cast alloy was subjected to warm working at 300 ºC and intercritically annealed at 580 ºC in two phase solid-liquid region. The result revealed accelerated spheroidization of the primary α- phase. The size of the spheroids varied from 45 to 60 µm. Analysis of wear track surfaces brought out a clear evidence of co-existing abrasion and adhesion mechanisms of material removal during tribological study of SIMA processed alloy.

In centrifugal casting the optimum mould speed was found to be 1980 rpm as it produced fine microstructure in the casting resulting in optimum hardness, ultimate tensile strength and wear rate.

Scope of the Future Work

- SIMA process of the Al-10Cu alloy can be studied by varying the semi-solid temperatures and deformation percentages.
- Rheocasting of Al-10Cu alloy can be studied by producing at different semi-solid pouring temperatures and stirring time.
- The effect of liquid droplets distribution in the solid matrix on the mechanical and wear properties of other alloys can be studied
- An attempt can be made to produce semi-solid centrifugal casting for achieving good mechanical and wear properties.