Influence of soaking time of modifier in melt on microstructure of Al-12%Si alloys

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Abstract. The influence of the soaking time of the silumin melt with a modifying nanopowder W on the microstructure and the impact toughness of the Al-12%Si silumin is studied in the paper. It was established that when introducing 0.1 mass. % of W and soaking it for 10 min, up to 30% of the inassimilable nanopowder W remains in the crucible after pouring. At that, the microstructure of castings is refined, and the impact toughness increases by 12 % compared to the initial alloy. It was established that the powder remaining in the crucible after melting consists of the remnants of metallic W, WO₃ and W₃O. Nanopowder W is completely assimilated by the melt if the soaking time is increased by 120 min; in this case, the impact toughness reduces, which is connected with the increase of dendrites and silicon wafers sizes.

1. Introduction

At present, silumins are one of the most wide spread alloys among non-ferrous metals. Silumins are widely used in aircraft, shipbuilding, automobile production and for daily living needs. Their wide application is caused by their increased fluidity, low tendency to shrinkage formation during pouring, a possibility of soldering and welding, low cost. Eutectic and hypereutectic silumins possess enhanced foundry properties because of low shrinkage and high fluidity. Eutectic silumins contain about 12 % Si and at room temperature, they consist of eutectic – a mixture of the solid solution of aluminium containing up to 1.65 % Si and pure silicon wafers. This type of the phase composition of eutectic silumins in the solid state determines their low mechanical properties. Low mechanical properties of eutectic silumins limit the field of application of this material.

To enhance the mechanical characteristics of silumins is possible owing to [1, 2]:
1) changing the morphology and reducing the crystals sizes of primary and eutectic silicon.
2) refining structural components of the alloy including predominantly ferrous-based intermetallicides insoluble in matrix.
3) inhibiting dendrite crystallisation by means of dispersion and transformation of dendrites of the solid solution into equiaxed crystals.

One of the efficient ways to realise these mechanisms is an increase of the number of nucleation centres during the crystallisation process by means of modifying silumins. A widespread method influencing the uniform formation of the microstructure and enhancement of strength properties of Al-Si alloys is modification of super- and nano-dispersed particles of different chemical compositions [3-7]. The basic nanodimensional modifiers used for silumins are the powders of metal oxide and nitrides [3, 4, 8]. They possess high melting temperature and high inertia, which allows them not to dissolve in the melt and to serve as crystallisation centres. The particles of refractory metals (W, Ti, Mo and others) act in a similar way. When introducing the powder particles into the melt, it is necessary to provide their wettability in order to achieve the modification effect.

The effect of the particles of chemical compounds differs from the effect of pure metals. Metals of powders (Ti, Mo, Cr, Sb etc.) interact properly with the silumin melt [9]. According to the state diagram of Al-W, the solubility of W in the α-Al solid solution amounts to 0.16-0.20 mass. % [10]. Therefore, the particles of nanopowder W, introduced into the melt in the amount of up to 0.5 mass.
%, will dissolve completely in the α-Al solid solution. In the process of formation of silumin structures, refractory wolfram particles being additional crystallisation centres will influence the refinement of structural components of castings.

This paper is aimed at the study of the influence of the soaking time of the silumin melt with the modifying nanopowder W on the microstructure and the impact toughness of the Al-12%Si alloy.

2. Methods and materials
The Al-12%Si alloy was chosen as a material of the study. The pig silumin, manufactured at a metallurgical plant, with the chemical composition according to Table 1 was used as a mixture for melting.

| Table 1. Chemical composition of Al-12%Si alloy used in this work (mass. %) |
|-----------------|-------|------|------|-------|------|-------|------|-------|------|
| Si              | Fe    | Cu   | Mn   | Zn    | Ti   | Mg   | Ca   | Pb    | Ni   |
| 11.2            | 0.44  | 0.29 | 0.28 | 0.08  | 0.04 | 0.05 | 0.04 | 0.01  | 0.03 |

The wolfram powder obtained by the electric blasting method with an average particle size of 240–380 nm and a specific surface area of 1.7-2.4 m²/g was used for alloy modification. The average size of wolfram particles amounts to $d_{av} = 1.32$ μm. On the surface of wolfram particles, there are nanoparticles, the average size of which is $d_{av} = 0.07$ μm (Figure 1).

![Figure 1](image1.png)

**Figure 1.** Nanoparticles on tungsten particles surface (a) and histogram of nanoparticles distribution by sizes (b).

Silumin melting was carried out in a muffle furnace. For speeding up the melting, the furnace was, first, heated up to 800°C, and then a steel crucible with a batch of the mixture was placed into the furnace. The mass of one melting amounted to 500 g. The mass of the charged metal and the mass of the powders intended for modification were measured by means of an analytical balance ‘Shinko HJR-620CE’ with an accuracy of up to 0.01 g. After silumin melting, the crucible was withdrawn from the furnace, the mirror of the melt was cleaned from the oxide film and then the modifier was introduced. The amount of the introduced modifier was 0.1 mass. %. After the introduction of the modifier, the melt was soaked in the furnace at a temperature of 800°C during 10, 60, 120 min. The pouring was carried out at a rate of 0.06-0.09 l/s. Per each soaking period, there were 3 meltings to accumulate statistics.

Granulometric analysis of nanopowders W was conducted by the method of laser diffraction (particle analyzer ‘SALD-7101’). The chemical and phase compositions of the wolfram powder were studied by the methods of optical emission spectroscopy (PMI-MASTER Sort) and X-ray diffraction analysis (XRD-7000S). The chemical analysis of silumin is determined by an average value of five measurements. Before measuring, the surface of samples was milled at a depth of 5 mm. The measurement error of the device amounted to 0.01%.
3. Results and discussion
The results of the conducted experiment showed that after conducting the melting and soaking of the melt containing the powder in the furnace for 10 min at 800 °C, 25-30% of the powder remains on the walls of the crucible after pouring of the melt. The increase of the soaking time of the crucible with the nano-powder, introduced into the melt, up to 60 min results in the fact that the amount of the powder remaining in the crucible after pouring reduces to 10-15%. When increasing the soaking time to 120 min, no powder remains on the crucible walls.

The studies showed that the powder, remaining on the crucible walls after melting, differs from the initial one by the composition. According to the data of XRD, the initial wolfram powder contains metallic wolfram (85 vol. %) and wolfram oxide W3O (15 vol. %) (Figure 2 a). According to XRD, the powder remaining in the crucible after melting consists of the remnants of metallic W and its oxides of different modifications (WO3, W3O) (Figure 2 b). After melting, the volume fraction of the metallic W amounts to 72 vol. %, W3O – 8 vol. % and WO3 – 20 vol. %.

![Figure 2. X-ray pattern of nanopowder W: a – initial state; b – remaining in crucible after melting (soaking of the melt containing powder in the furnace during 60 min).](image)
The variation in the time of soaking of the modified melt in the furnace leads to a fairly significant change in the microstructure. A ten-minute soaking of the melt significantly refines dendrites of the solid solution. Short axes of the first order with a length of less than 200 µm and short axes of the second order (with a length of not more than 30-40 µm) are formed. Silicon wafers are subjected to significant refinement. When the melt with the nanopowder W is soaked in the furnace for 60 min, developed dendrites of the solid solution of silicon in aluminium are formed in the castings structure. According to the data of optical microscopy (Figure 3 c), it is obvious that the dendrites of the solid solution have developed axes of the first order of a 500 µm length, as well as quite developed axes of the second order. When increasing the soaking time up to 120 min (Figure 3 d), the length of the first-order axes reduces substantially. Similarly, the length of the second-order axes reduces as well, and they become less developed (of less length and thickness). Changes occur in the silicon wafers. Thus, the increase of the soaking time in the furnace up to 120 min reduces the average length of silicon wafers by 15-25%. In this case, the thickness of wafers increases.

Figure 3. Microstructure of Al-12%Si alloy: a – initial state, soaking time is 10 min; b – 0.1 mass. % W, soaking time is 10 min; c – 0.1 mass. % W, soaking time is 60 min; d – 0.1 mass. % W, soaking time is 120 min.

These variations in the structure affect the properties significantly. The performed testings of samples obtained during different soaking periods with the introduced modifier in the furnace demonstrated a significant influence on the impact toughness (Figure 4). These data demonstrate that the impact toughness of unmodified samples amounts to KC 15.6. Addition of the powder and soaking of the melt with the powder in the furnace during 10 min facilitates the increase of the impact toughness up to KC 18. A further increase of the soaking time of the melt in the furnace leads to the reduction of the impact strength up to KC 13.4 (Figure 4).
4. Conclusion
On the basis of the carried out experiment, it is possible to conclude that the introduction of the nanopowder W into melt Al-12%Si produces a significant modifying effect and allows changing the microstructure. In this case, a significant influence on the effect of powder modification will be exerted by the time of soaking the alloy with the nanopowder in a heated state. In case of the nanopowder introduction, soaking during 10 min and the pouring of the melt, up to 30 % of the inassimilable powder remains in the crucible. It is possible to avoid it by increasing the presence time of the melt with the powder in the crucible up to 120 min.

The soaking of the melt with the modifier during 10 min followed by further pouring is optimal from the viewpoint of impact toughness. This is in good agreement with the data of metallographic analysis. Samples, obtained during a 10 min soaking period, possess refined dendrites with an undeveloped structure and short silicon wafers. The increase of the melt soaking period in the furnace leads to the increase of the dendrites and silicon wafers sizes even in comparison with the unmodified structure. Therefore, the impact toughness of these samples reduces.

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