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Technical note

The preparation of Al-Si cast alloys specimens for TEM observations

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Abstract. — An original method for preparing thin foils from Al-Si cast alloys is described. This simple method consists of one – jet electropolishing accompanied with ultrasonic "cleaning". The aim of this ultrasonic bath is to remove eutectic Si precipitates forming a specific "skeleton" in the specimen. This framework hinders the process of polishing and makes TEM observations more difficult, especially when high angle tilting is used.

Most TEM observations are performed on one-phase materials or specimens where the second or more phases are highly dispersed and/or relatively small in size (e.g. carbides, nitrides etc.). Very few TEM studies concern casting alloys [1, 2]. The reason is enormous difficulty in preparation of good quality thin foils either by chemical or electrochemical polishing methods. Sometimes it can be overcome by using ion-milling but this needs a lot of time and the results are not guaranteed. There are at least two reasons why the first methods of low efficiency. First of all, cast alloys are usually of eutectic composition. It means that the specimen consists of at least two phases of comparable contribution. In most cases these main constituents differ from the point of view of their chemical properties. It means that it is practically impossible to find the composition of a thinning solution dissolving each phase with the appropriate rate. The other reason concerns the specific distribution of the second phase.

Let us consider near eutectic Al-Si cast alloys. Their structure consists of more or less dispersed Si precipitates forming a specific skeleton inside the relatively soft α-solid solution. This α phase is dissolved while thinning with the solution for electropolishing Al and its alloys. However, the continuous skeleton of eutectic Si precipitates is not dissolved and remains as a characteristic "scaffolding". It thus prevents the dissolved Al-atoms to go away from the region near the surface of the thinned specimen. This in turn disturbs the conditions appropriate for polishing and causes etching or pitting. Such a rigid skeleton makes impossible to catch the moment of perforation while thinning with the jet electropolisching method.

In this letter, a preparation method for electropolishing Al-Si type cast alloys is proposed. The procedure is tested on a AlSi9 (Mg) cast alloy. The rod-shaped 3 mm diameter specimens are cut from the casting and sliced into 0.1 mm thick discs using wire saw type IF-07A. These discs are electropolished using the one-jet method and the Hacking electrolyte with the composition [3]:

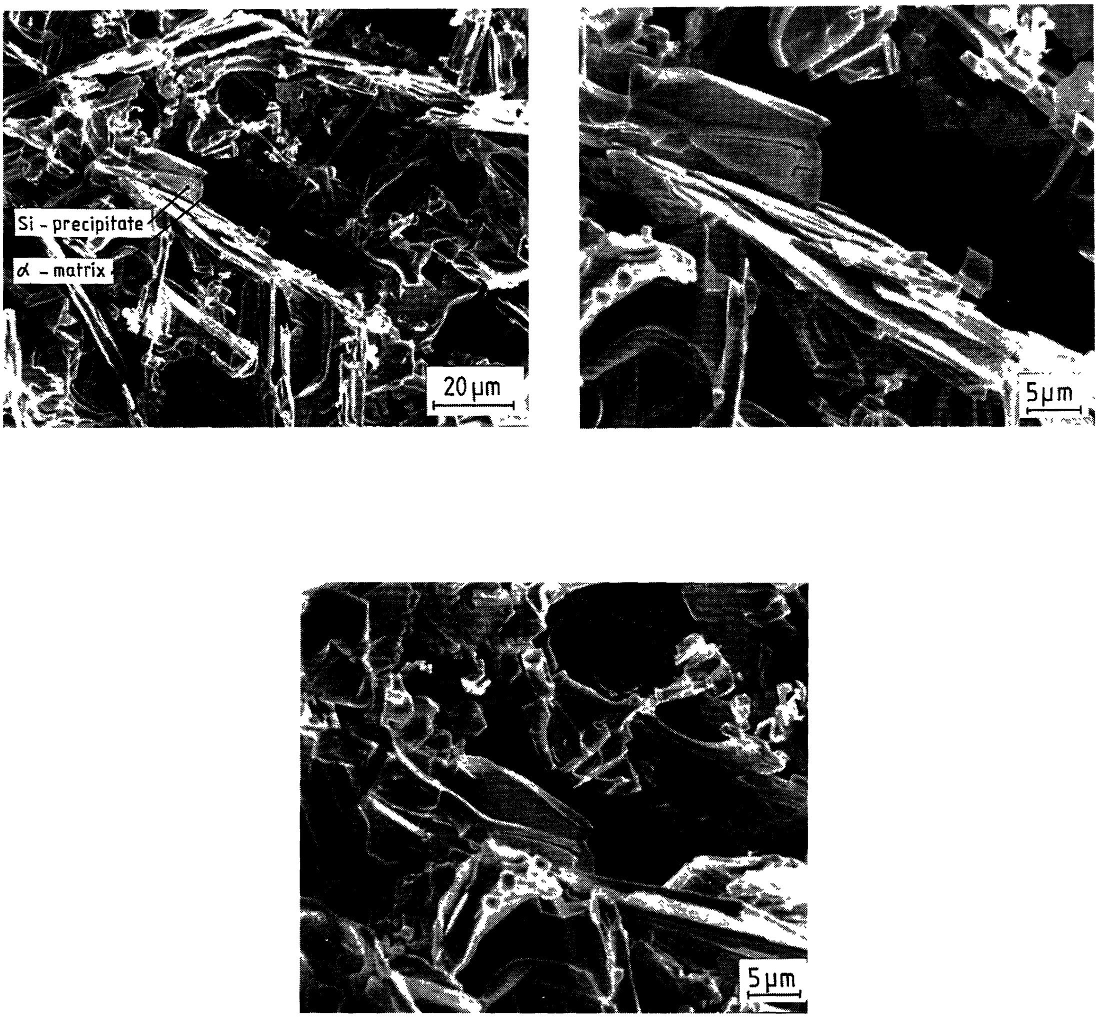

Fig. 1. — The "skeleton"of eutectic Si precipitates on the surface of electropolished AlSi9 cast alloy specimen: a) ×750; b) ×2000 without tilting, c) ×2000 The same region but after 30° tilting.

acetic acid (CH_3COOH-glacial) – 400 ml,
orthophosphoric acid (H_3PO_4) – 300 ml,
nitric acid (HNO_3) – 200 ml,
water (H_2O) – 100 ml.

The diameter of the jet is 1.1 mm. The voltage and current are $U_1 = 30$ V and $I_1 = 60$ mA and $U_2 = 45$ V and $I_2 = 90$ mA for preliminary and final polishing respectively. Some of the foils are polished traditionally and the others according to the method elaborated by the author, in which an ultrasonic bath is used for removal of Si brittle precipitates: the discs are first polished traditionally for approximately 30 seconds and then placed into alcohol for 10-15 s ultrasonic washing. After this treatment the foils are electropolished again. This procedure is repeated three times for one side of the specimen. The laquer is deposited on the already polished surface. After its solidification the opposite side is polished exactly the same way until the perforation appeared.

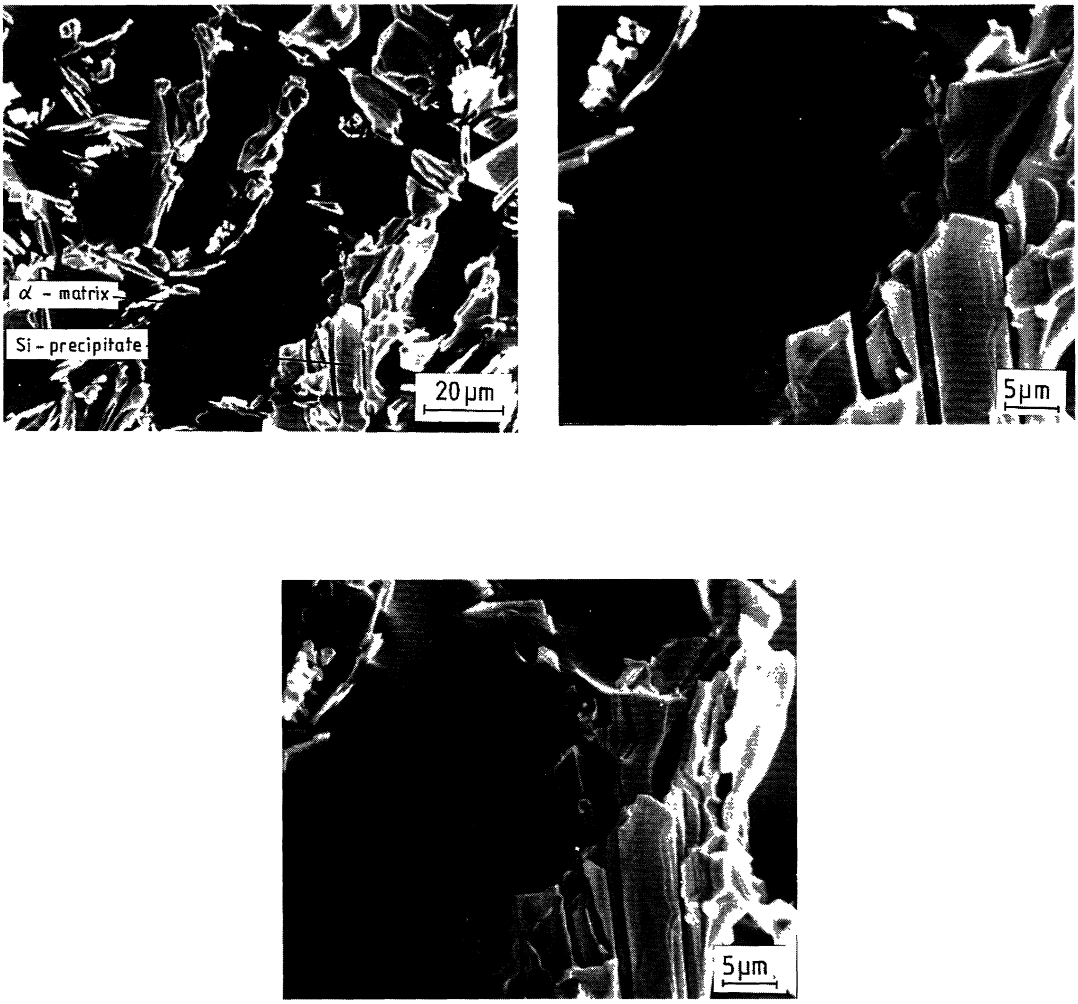

Fig. 2. — The surface of thinned AlSi9 cast alloy prepared according to the new methode: a) ×750; b) ×2000 without tilting; c) ×2000 the same region but after 30° tilting.

Figures 1 and 2 show the surface of the specimens prepared according to the conventional (Fig. 1) and the new method (Fig. 2). The first of them presents the surface observed at relatively small magnification (× 1000) with a SEM microscope. As mentioned above a dense skeleton of eutectic Si precipitates is clear visible. The micrograph (Fig. 1b) shows a dark region being one of the arm of α-solid solution dendrite. This region is observed at magnification ×2000 without tilting of the specimen. The same area but after 30° tilt is shown on figure 1c. It is easy to see that α region has almost disappeared because it is shielded with Si precipitates.

The surface of the specimen of the same cast alloy prepared according to the new method is shown on the figure 2. The first picture is recorded at small magnification (× 750) when the surface is positioned perpendicular to the SEM axis. It is clear that the α-solid areas are of the same size as before (Fig. 1) but no Si skeleton is visible. Only small Si precipitates are observed. They are distributed in the interdendritic space but they form no plates or rosettes. The next picture (Fig. 2c) shows the same region after 30° tilting in which condition the α-solid solution remains rather well visible. Figure 3 shows an example of TEM micrograph taken from the specimen prepared according to the method elaborated with the author. On this picture the microstructure of the precipitation hardened AlSi9(Mg) cast alloy is presented. The dark-non transparent regions at the left corner are relatively big eutectic Si precipitates, while the white region corresponds to the α-matrix. In this α-solid solution area the metastable needle-like β" precipitates pinned at the dislocations are clearly visible. As it is well known, they can be observed when some specific diffraction conditions are fulfilled [4]. This in turns requires the tilting of the specimen which is accomplished in the goniometer stage. In the case of the micrograph shown in figure 3, the tilting angle of the thin foil was approximately 20 degree. In spite of this tilting the microstructure of the AlSi9(Mg) cast alloy is well revealed.

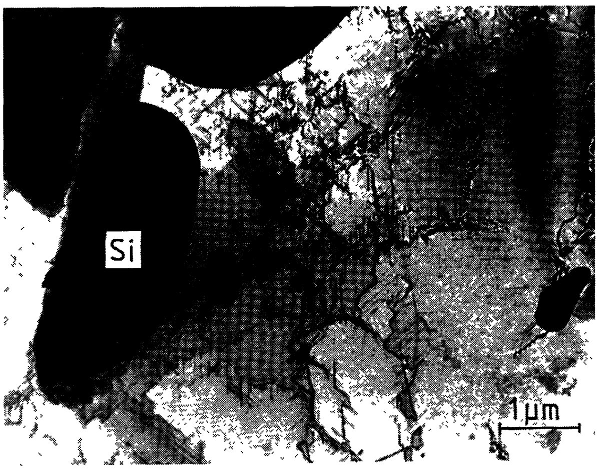

Fig. 3. — The TEM micrograph showing the structure of the precipitation hardened AlSi9(Mg) cast alloy.

It seems that the proposed method could substantially improve TEM studies of casting alloys. However there are some limitations for it. One condition is that the second phase has to be relatively brittle. The second one concerns the purpose of TEM observations. This method can be applied to the study of precipitation processes, phase identification, grain and subgrain structure etc. with no limitation. On the other hand we have to be careful while studying dislocation structures because they might be influenced by the stresses generated in the matrix-precipitate interface.

References

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