Elaboration of AlSi13 Casting Alloys modified using Directional Solidification Processing

Elaboration de l'alliage AlSi13 modifié et solidifié unidirectionnellement

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ملخص

لتحسين الخصائص إن الخصائص الميكانيكية لسبائك AlSi13 ترتبط بالأبعاد و بالشكل و توزيع Si eutectique و كذلك بكمية Si الأولي الموجودة في البنية الداخلية للمادة. عدة معالجات و طرق مقترحة كاللتغيير . في هذا العمل قمنا بدراسة مفعول التغيير بنسبة 1% و 2% من المعدل المركب من NaCl+45%NaF+15%KCl 40% مدموج بالترسيخ السريع لسبائك AlSi13 بتقنية بريدج مان. و بدالك لاحظنا انخفاض المسافة بين التغصن (شجيري ثانوي) و هذا خاصة بالنسبة 2% من المعدل عند سرعة السحب المنخفضة . كما لوحظت أن اتجاه ات التغصنات تفضل اتجاه السحب عندما تكون السرعات منخفضة آما الصلادة HB ومقاومة الهشاشة فهي أحسن.

الكلمات المفتاحية : AlSi13 - Si eutectique سبائك -تقنية بريدج مان -السلسيوم الأتكتيكي

Abstract

In order to improve the mechanical properties of Al-Si13 alloys various treatments and procedures are recommended such as modification treatment of the molten alloy.

In this work, the effects of the modification of the alloy composition owing to the addition of 1 wt.% and 2 wt.% modifier composed of 40 wt.% NaCl and 45 wt.% NaF and 15 wt.% KCl, combined with the application of unidirectional solidification by means of the Bridgman type were studied on the Al-Si13alloy. Microstructural analyses revealed a decrease in the interdendritic spacing(SDAS), in particular owing 2% of the modifier prepared with a reduced pulling traction. The dendrites were preferably oriented along the pulling velocity for fairly low pulling velocity (500 µm/s). These microstructural modifications enabled an increase of both the hardness (HB) values and the impact toughness.

Key words: AlSi13 alloy, Modification treatment, Directionally solidification, Bridgman technique, Silicon eutectic.

Résumé

Afin d'améliorer les propriétés mécaniques des alliages AlSi13 divers traitements et procédures sont préconisés tels que la modification.

Dans ce travail, les effets de la modification avec 1 wt.% et 2 wt.% d'un modificateur composé de 40% NaCl, 45% NaF et 15% KCl combiné à une solidification unidirectionnelle de type Bridgman sur un alliage AlSi13 ont été étudiés. L'analyse microstructurale a révélé une diminution de la distance interdendritique (SDAS) notamment avec 2 wt.% du modificateur et une vitesse réduite du tir (500 µm/s). Les dendrites sont orientées préférentiellement le long de la direction de tir pour des vitesses de traction assez faibles. Par contre, les valeurs de dureté (HB) et de résilience des échantillons solidifiés se sont accrues.

Mot clé: AlSi13, Modification, solidification unidirectionnelle, technique de Bridgman, silicium eutectique.

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1. Introduction

Aluminum and its various alloys, particularly those containing silicon as a major constituent element, are currently experiencing considerable growth, which has resulted in a high degree of reliability in a wide range of applications, in particular in the fields of aerospace and transportation. Thanks to their excellent weight-to-resistance ratio, their ease of shaping and their good resistance to corrosion, aluminum-silicon (Al-Si) alloys constitute an important class of materials used for the manufacture of engine parts and cylinder heads [1-2]. Despite these advantages, numerous studies have shown that these alloys when elaborated by casting process are sensitive to defects, which strongly affect the metallurgical quality of the product and consequently the mechanical behavior especially the resistance to fatigue [3-4]. These defects of various natures, which depend to a large extent upon the chemical composition (alloying elements) but also on the elaboration conditions (melting, liquid temperature treatment, modification) and solidification, have a direct impact on the microstructure. Particularly, the spacing of the secondary dendrite arms (SDAS), the porosity, the presence of oxide films, the size and morphology of Si phase in the eutectic, and the shape as well as the distribution of intermetallic compounds and micro-segregations, which are among the most important factors that must be controlled in order to deliver sound parts conforming to the required quality [5-6].

In order to optimize the mechanical characteristics of the parts, various treatments and casting techniques are currently used such as modification treatment of the molten alloy and directional solidification [7-9]. On one hand, these modifications enable a reshaping of the eutectic silicon from acicular to fibrous by adding of elements such as sodium, strontium, etc., to suppress the polyhedral plates of the primary silicon and also to reduce the macro-shrinkage into micro-shrinkage that is less harmful from the mechanical property standpoint [8]. On the other hand, the size of the grains, the volume fraction of micro porosities, the inclusions and the formation of the various intermetallic phases are directly related to the cooling rates that influence the morphology of intermetallics, too. It is now acknowledged that the interdendritic spacing, size of silicon phase and eutectic morphology have positive effects on the properties of the alloy while the volume fraction of porosities and intermetallics, P-AlFeSi phase have deleterious effects [10-12].

This study was undertaken to examine these questions, and this article presents the results of two combined effects of modification treatment of the molten alloy by addition of a salt and processing condition on the microstructure and mechanical properties of a unidirectionally solidified Al-Si 13 alloy. The modification of the Al-Si13 alloy composition was examined by addition of Na element in the introduced in the form of salt introduced into the melt at different concentrations (1 and 2%). The processing condition was the pulling traction velocity during solidification by using the Bridgman technique and particularly the spacing of the secondary dendritic arms (SDAS).

2. Experimental Procedure

We used as starting materials standard ingots of the eutectic Al-Si13 alloy obtained from the National Company for Industrial Vehicles (NSIVGroup) in Algeria. The chemical composition of this alloy according to NFA57-702 is given in Table 1.

The ingots were elaborated in an electric furnace with a removable graphite crucible resistor. The alloy was first modified by means of addition of different concentrations (1% and 2%) of a salt in the form of flux (40% NaCl, 45% NaF and 15% KCl) [11]. The salt was prepared and dried in an oven then introduced at the surface of the bath at a temperature of 730 °C. After 10 minutes, a scrubbing operation was carried out and the metal, whose temperature was controlled thoroughly by a thermocouple, was poured at a temperature of 720 ºC. The ingots obtained are 120 mm in length and 180 mm in diameter.

The alloys were then re-melted and unidirectionally solidified. For that purpose, the specimens (180 mm in diameter and 120 mm in length) were sealed in quartz tubes and placed in the furnace of a vertical Bridgman directional solidification apparatus [12] The samples were heated for 15 min at 850 °C before being moved downward at a constant traction rate of either 500 or 4000 µm/s into the cooled water reservoir. A fine Pt-Pt10%Rh thermocouple protected by alumina sheaths embedded in the furnace was used to measure the temperature gradient, which was varied from 10 to 14 °C/min.

For microstructural analyses, the longitudinal sections of the samples were polished and etched with a 2.5%HCl–1.5%HNO_3–1%HF reagent for 10 to 30 s [5]. The specimens were deep etched by suspending the polished area in an ultrasonic bath with the etchant for 10 min. Values for the SDAS were obtained from averaging a minimum of 10 observations by means of a JEOL 5800LV secondary electron microscope. Brinell hardness (HB) measurements were carried out on selected areas of the longitudinal sections using a hardness tester and the values were averaged on a minimum of 10 measurements. Charpy tests were performed 10.8 x 8.2 mm^2 samples for evaluation of the fracture energy.

3. Results and Discussion

3.1. Influence of the modification on the microstructure of AlSi13

The microstructures of the sections of the as-cast and un-modified alloys prepared in sand and metal molds are shown in Figure 1, while those of the modified and directional solidified alloys are shown in Figures 2 and 3. Figure1 reveals optical micrographs showing longitudinal section of Al-Si13 alloy: (a) without modification cast in sand mold, (b) cast in metal mold, (c) Al-Si13 alloy modified with addition of 1% (NaCl+NaF+KCl) in sand mold and (d) Al-Si13 modified with addition of 2% (NaCl+NaF+KCl) in sand mold. Figure 2 reveals SEM micrographs showing the structural refinement obtained by modification depending to the salt percentage: (a) 1% salt with v = 500 µm/s; (b) and(c) 2% salt with v = 500 µm/s. Figure. 3 shows optical micrographs of longitudinal section of Al-Si13 alloy without modification obtained after unidirectional solidification: (a) v = 500 µm/s, (b) v = 4000 µm.

These micrographics reveal for all conditions the usual structure of the Al-Si13 alloy consisting of the solid solution α-Al, an acicular, lamellar or globular ($\alpha_{Al}+\beta_{Si}$) eutectic depending on whether the alloys have been modified or not cast in a metal or sand mold, and polyhedral crystals of the primary silicon phase characteristic of the silicon containing alloys.

In fact, the four structures shown in Figure 1exhibited an acicular structure containing dendrites of the solid solution α aluminum, ($\alpha_{Al}+\beta_{Si}$) in the form of rather large lamellae and platelets of primary Si, which is commonly observed for raw Al-Si alloy prepared by sand casting method when the alloy has not undergone any treatment either in the liquid (refining or modifying) or solid state. This type of microstructure is very unfavorable on a mechanical properties because it tends to result in hard and brittle alloys that are difficult to machine. For this reason, this kind of microstructure must undergo a modification treatment intended to refine the grains and/or to transform the morphology of the eutectic from granular to fibrous, and in addition aimed at suppressing the polyhedral plates of the primary silicon phase. In the Figure 1b, which shows the microstructure of an as-cast alloy obtained in a metal mold, the same constituents are noted, though much finer, but always containing smaller primary silicon grains due to an increase in the number of germs during crystallization resulting from a very rapid cooling, which limits the growth rate of dendrites and lamellae. Figures 1c and 1d show the structures of the alloy modified with respectively 1 and 2% addition of salt in the form of flux.

The examination of these structures revealed a significant morphological change in the eutectic silicon resulting from the salt addition in the molten Al-Si13 alloy. The initially squared silicon phase in the eutectic appeared in the form of fibers and finer than that of the metal mold casting as illustrated by the micrographs of the electron microscope in Figure 2. Thus, the structure 1c of the 1% modified alloy reveals a completely modified eutectic and the total absence of the polyhedral grains of the primary silicon phase. This phenomenon is in principle due to the poisoning of the sites used for the growth of this phase by owing to the covering by a (Na_2Si) during solidification, which makes it difficult to appear [13-14]. In Figure 1d, on the other hand, for the 2% modified alloy, we notice a structure that is always modified but with the presence of lines of sur-modification and defects (inclusions and porosities) characteristics of a sur-modified state. The appearance of this aspect in the microstructure is rather harmful because it greatly affects the metallurgical quality of the alloy by the reduction in particular of the sealing (gassing phenomenon) of the parts and the reduction of the values of the mechanical characteristics.

Figures 3a and 3b shows the structures of the alloy directionally solidified at, respectively, two pulling velocities of 500 and 4000 µm/s in the absence of modifier. The acicular structure consists of the AlSi eutectic preferably oriented along the pulling direction that develops along the heat flow under low pulling velocities (i.e., around 500 µm/s). For larger velocities (i.e., v = 4000 µm/s), the volume fraction of eutectic increased and was found to be finer.

Figure 4 reveals optical micrographs of longitudinal section of Al-Si13 alloy modified and obtained after unidirectional solidification: (a) Al-Si13 modified with 1% salt & v = 500 µm/s, (b) Al-Si13 modified with 1% salt & v = 4000 µm, (c) Al-Si13 modified with 2% salt & v = 500 µm/s and (d) Al-Si13 modified with 2% salt & v = 4000 µm.
Figures 4c, 4d, 4e and 4f show the microstructures of the alloy containing 1% and 2% modifier produced by directionally solidification. Even during such processing condition, the microstructure of the alloys prepared with both pulling velocities of 500 and 4000 µm/s preserved the modification of the microstructure characterized by the presence of a fibrous eutectic fibrous. For both alloys containing 1% and 2% modifier, the condition corresponding to the low pulling velocities provided a better orientation of the dendrites of the αAl phase, with the growth of the dendrites preferably oriented. For larger velocities, i.e., v = 4000 µm/s, the α-Al dendrites grew parallel to the cooling direction. The refinement of the microstructure can be illustrated by the interdendritic arm spacing.
Figure 5 reveals Influence of the % of modifier and the traction velocity on the SDAS.This Figure indicates that the measurements of the spacing between the secondary dendrites (SDAS) decreased as a function of the pulling velocity and that a finer microstructure can be achieved particularly in alloys containing 2% modifier. As the pulling velocities increased from 500 µm/s to 4000 µm/s, the value of SDAS was respectively decreased from 28.32 ± 0.72 µm to 27 ± 1.6 µm, for 1% salt, and from 16.42 ± 0.25 µm to 8.92 ± 0.81 µm, for 2% salt. Apart from the dendrites described above, the microstructures of the alloy studied reveal the presence of the fibrous eutectic (αAl + βSi) distributed between the dendrites of the αAl phase, which confirms the persistence of the modification effect even after reheating owing to the addition of a sodium-based modifier.

The micrographs also indicate the absence of primary silicon particles in the microstructure, which is confirmed by the X-ray diffraction analyses shown in figure 6. Figure 6 reveals the elementary chemical analysis by spectrometry. The decrease in the volume fraction occupied by the silicon phase before and after the addition of the modifier is also indicated.

3.2. Mechanical properties

3.2.1. Influence of the salt addition on the hardness

HB measurements were carried out on different regions along the longitudinal sections of the modified and unmodified samples (Fig.7). Figure 7 indicates the influence of the rate of modifier on the hardness. For alloys prepared by conventional casting in sand mold, the results revealed an increase in the hardness values for the alloy modified with 1 and 2% salt in comparison to the unmodified Al-Si13 alloy. The addition of the 1% NaCl+NaF+KCl salt resulted in a mark increase of the hardness, though further addition till 2% indicated a slight decrease. It is known that unmodified alloys exhibit a structure with an undesirable acicular morphology due to its fragility. It acts as a stress concentrator, which acts as a notch by reducing resistance, ductility and fatigue resistance. On the other hand, a well-modified structure in which the silicon phase becomes globular gives better properties. However, the presence of lines under modification and especially surmodification is detrimental to the mechanical behavior of the alloy due to the presence of defects (cavities) and inclusions in the grain boundary affects the metallurgical quality of these alloys [15,16].

3.2.2. Influence of the pulling rate on the hardness

The results show that the variation of the hardness with the pulling velocity is not straightforward. In 1% modified alloy, the hardness is the largest when the alloy is prepared without pulling traction (i.e., v = 0 µm / s). As the pulling traction increased (v = 500 µm / s), the hardness first decreased by about 12 % to increased till value of about 63.1 ± 0.2 HB for v = 4000 µm / s. For alloy modified with 2% salt, the variations are of a reduced range however the alloy prepared without pulling traction is the harder while the one prepared with v = 4000 µm / s has an intermediate value. This increase as seen in

Figure 8 indicates the variation of the HB hardness for the un-modified and modified Al-Si13 alloy with the pulling rate.This figure is related not only to the modifying effect which gives fibrous morphology as mentioned above and in other works [17,18,19,20], but also at the applied cooling rates (pulling velocity), whose influence on the size of the grains, their morphology and those of the intermetallic phases and porosities is considerable [21]. This observation has been explained by the microstructures presented (Fig. 1, 2), where the increase in the cooling rate has favored in particular the reduction of the interdendritic spacing and the reduction in the particle size of the eutectic.

3.2.3. Toughness

Charpy tests were carried out on various samples of Al-Si 13alloy modified at 1% and 2% of salt. Figure 9 indicated that the Al-Si13 containing 1% salt displayed better toughness property than the alloy containing 2%. In addition, the highest value of the toughness was obtained for the alloy modified at 1% salt unidirectionally solidified at a pulling rate of 4000 µm/s. This demonstrates once again the beneficial impact of the refinement of the structure on the mechanical characteristics of these alloys.

4. Conclusion

Al-Si13 alloys were modified with addition of 1% and 2% salt composed of 40% NaCl, 45% NaF and 15% KCl. Samples were prepared by conventional cast methods in sand and steel mold as well as by directionally solidified solidification technique using two different pulling velocities, i.e., 500 and 4000 µm/s. The results of this investigation can be summarized as follow:

1 – The addition of NaCl-NaF-KCl salt induced a reduction of the interdendritic spacing distance (SDAS), in particular owing to 2% addition prepared under high pulling traction rate.

2 - The dendrites were found to be preferentially oriented along the pulling direction at low pulling velocities (500 µm/s).

3 – The preparation of the Al-Si13 alloys by unidirectionally solidification enabled a mark increase of the microhardness.

4 – The Al-Si13 alloys modified with 1% salt and prepared under unidirectionally solidification with the pulling velocity of 4000 µm/s displayed a best combination of mechanical property in terms of hardness and toughness. These properties were explained by the beneficial impact of the operations of modifying the directed solidification of the modification on the refinement of the structure these Al-Si13 alloys.

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Table 1. Chemical composition of the Al-Si13 alloy (wt.%)

Fe	Si	Cu	Mg	Mn	Ni	Zn	Sn	Pb	Ti
≤ 0.07	11-13.5	≤ 0.1	≤ 0.1	≤ 0.3	≤ 0.05	≤ 0.15	≤ 0.05	≤ 0.1	≤ 0.15

Table 2. Results of quantification phases by DRX of the Al-Si12 alloy (wt.%) without salt, and with 1% and 2% salt addition.

Echantillon	Volume Al (at.%)	Volume Si (at.%)	Al (wt.%)	Si (wt.%)
AlSi_0% salt	79	21	81,5	18,5
AlSi13_1% salt	81	19	83	17
AlSi13_2% salt	84	16	86	14

Error of the order of + 5%

Integration in psi from 0 ° to 50 °

Figure captions

Figure. 1: Optical micrographs showing longitudinal section of Al-Si13 alloy: (a) without modification cast in sand mold, (b) cast in metal mold, (c) Al-Si13 alloy modified with addition of 1% (NaCl+NaF+KCl) in sand mold and (d) Al-Si13 modified with addition of 2% (NaCl+NaF+KCl) in sand mold.

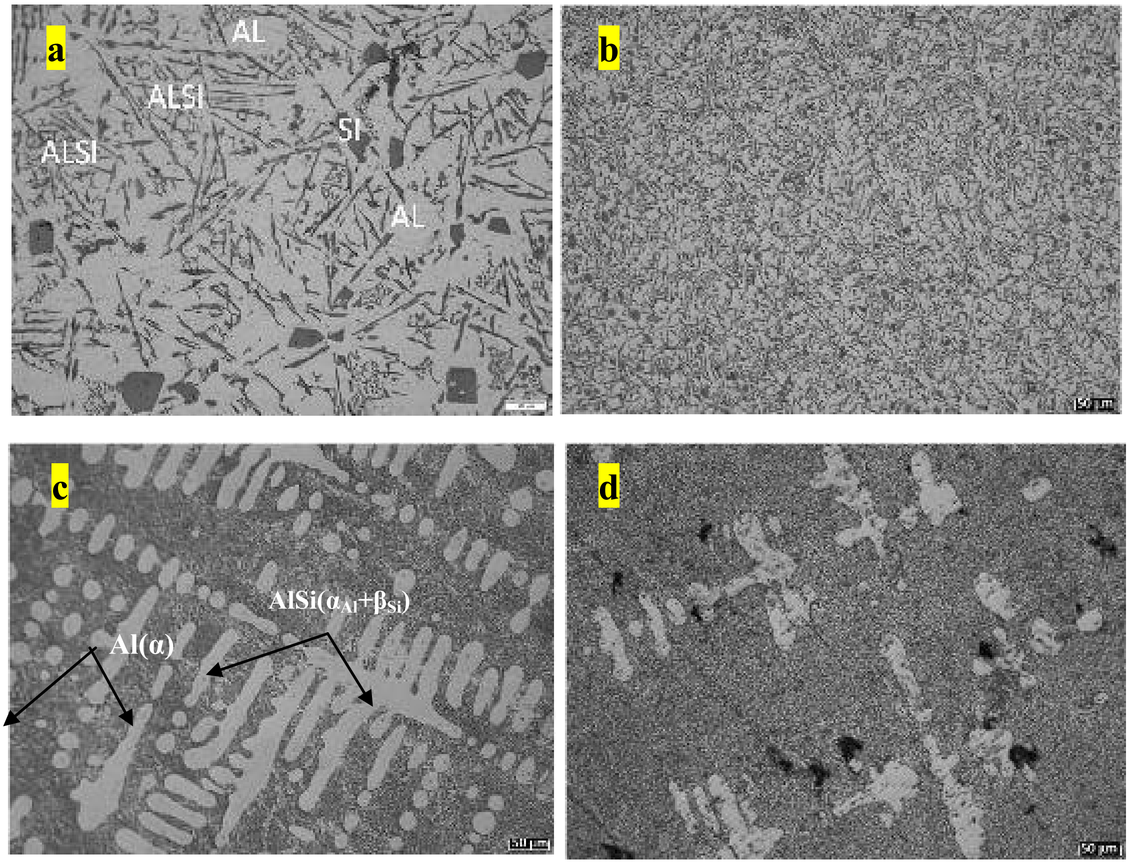

Figure. 2: SEM micrographs showing the structural refinement obtained by modification depending to the salt percentage: (a) 1% salt with v = 500 µm/s; (b) and(c) 2% salt with v = 500 µm/s.

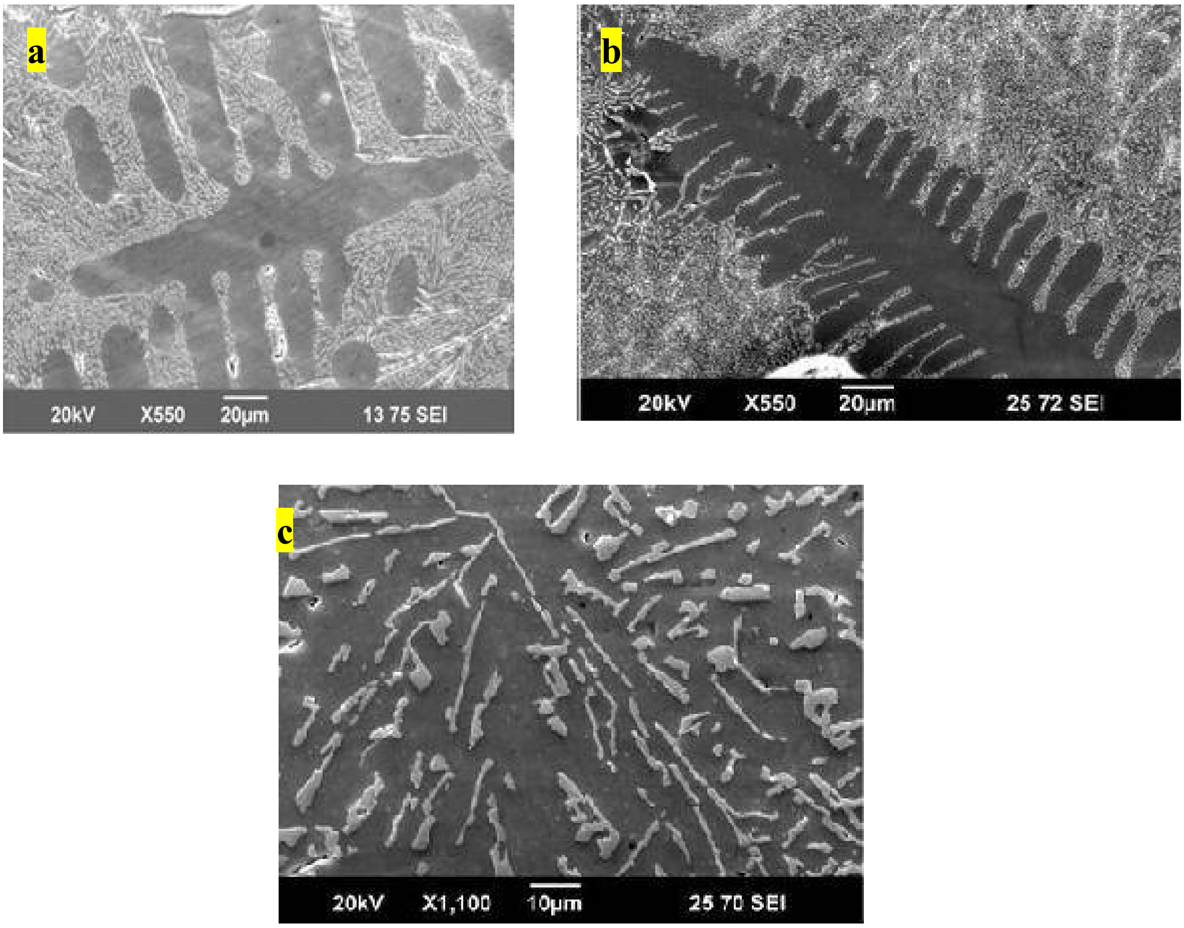

Figure. 3: Optical micrographs of longitudinal section of Al-Si13 alloy without modification obtained after unidirectional solidification: (a) v = 500 µm/s, (b) v = 4000 µm.

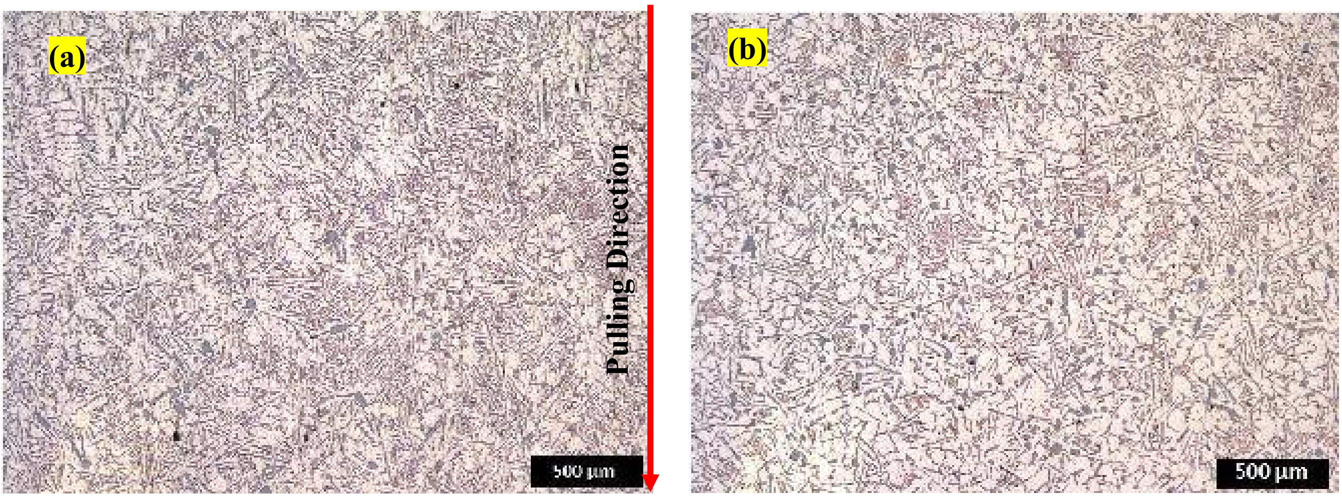

Figure. 4: Optical micrographs of longitudinal section of Al-Si13 alloy modified and obtained after unidirectional solidification: (a) Al-Si13 modified with 1% salt & v = 500 µm/s, (b) Al-Si13 modified with 1% salt & v = 4000 µm, (c) Al-Si13 modified with 2% salt & v = 500 µm/s and (d) Al-Si13 modified with 2% salt & v = 4000 µm.

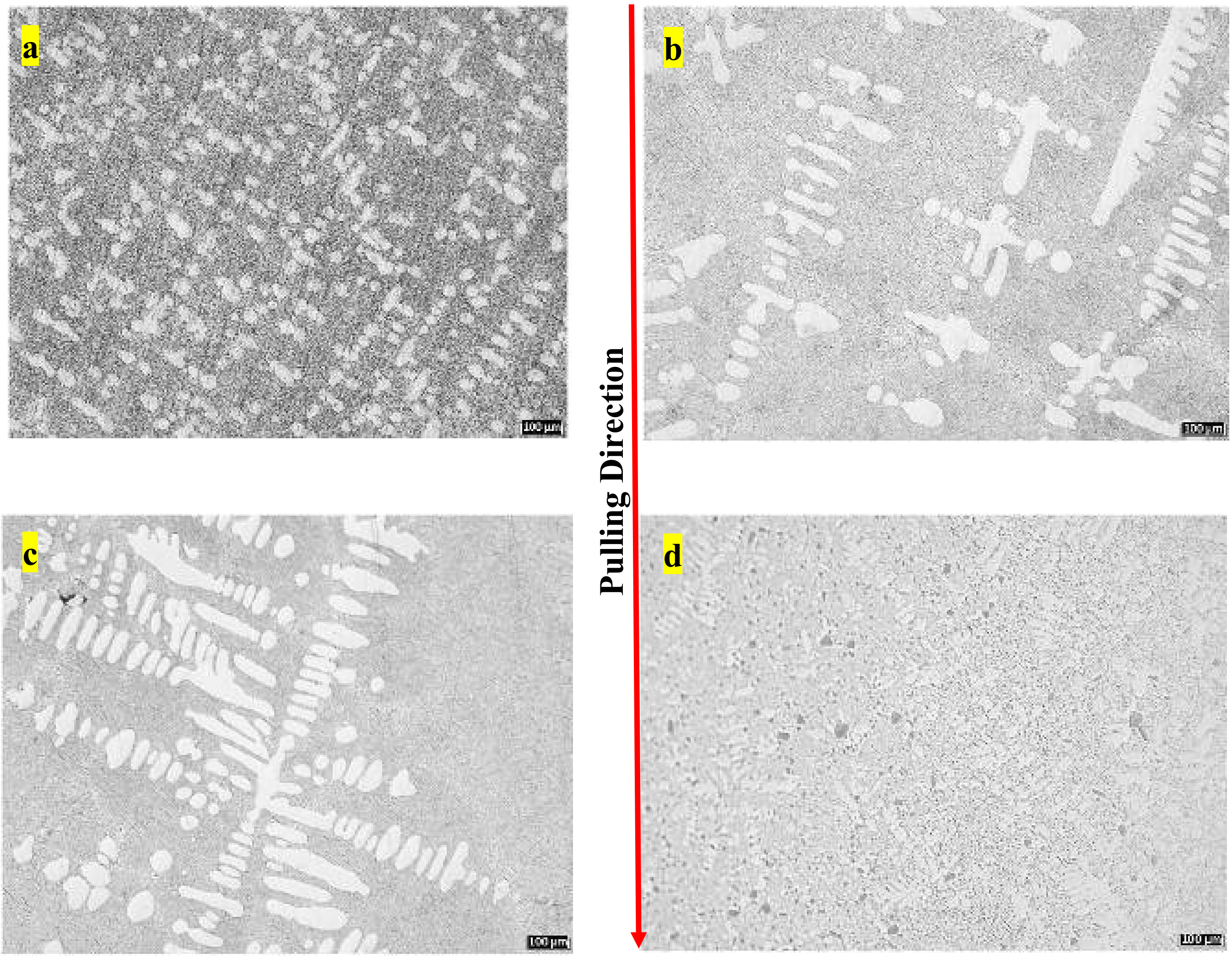

Figure 5: Influence of the % of modifier and the traction velocity on the SDAS.

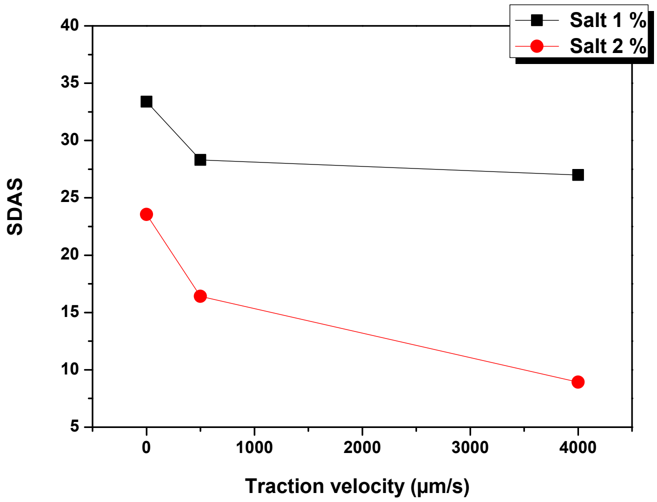

Figure 6: Elementary chemical analysis by spectrometry.

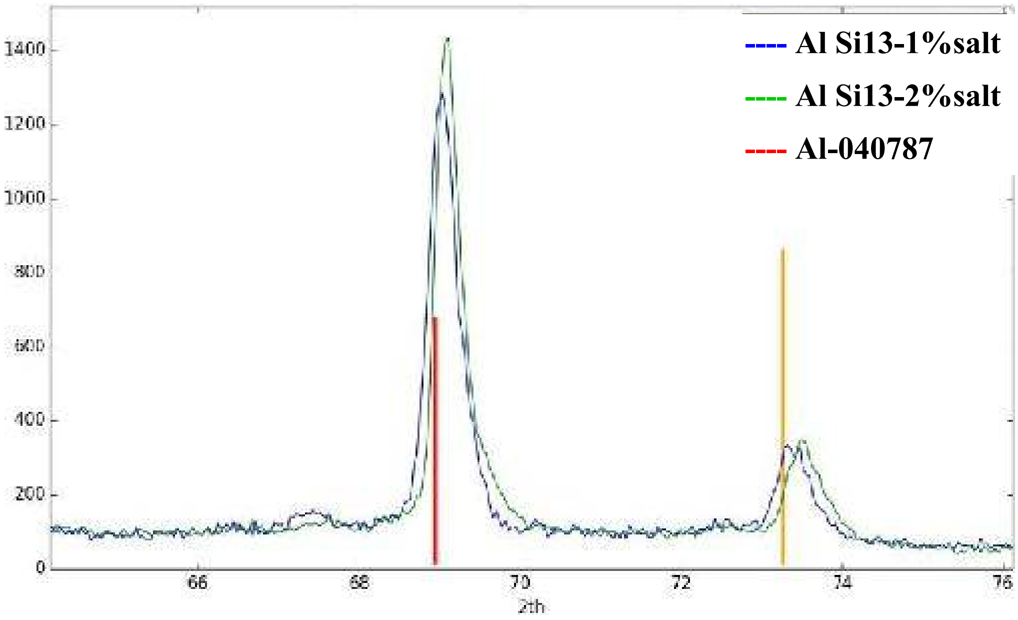

Figure 7: Influence of the rate of modifier on the hardness.

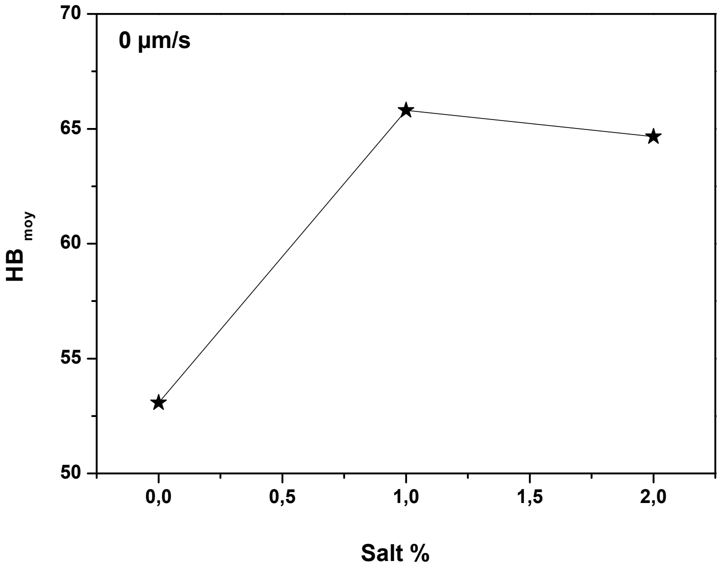

Figure 8: Variation of the HB hardness for the un-modified and modified Al-Si13 alloy with the pulling rate.

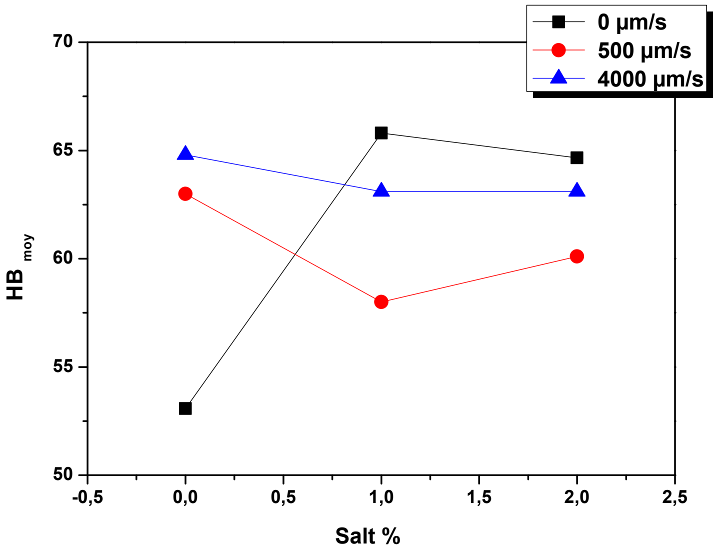

Figure. 9: Variation of the fracture energy measured by Charpy tests with the pulling rate for alloys prepared with different salt addition.