Effect of Squeeze Casting pressure on microstructure and mechanical properties of recycled aluminum alloy

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(Received: 23 November 2016, accepted: 25 December 2016)

Abstract: The present study investigated the effect of pressure variation during squeeze casting on the microstructural properties of recycled aluminum alloy. Our aim was to find a compromise between the microstructural and mechanical experimental results taking into account the effect of cooling rate. The results of microstructural observations by optical and scanning electron microscopy (SEM), showed a steady increase in the microstructural characteristics of the samples with the application of a 100MPa external pressure evidenced by a significant refinement of the grain size of the alloy. The mechanical analysis involved hardness (HV) and tensile tests showed an improvement with application of pressure under 100MPa. However, a sharp decrease observed with higher applied pressures up to about 150MPa, in that the grain growth and the high concentration of the linear and intrinsic defects on the alloy during molding caused the damage of the sample.

Keywords: Squeeze casting, Cooling rate, Microstructure, Mechanical proprieties.

1. Introduction

Aluminum recycling, particularly aluminum chips, has long been the object of thorough research for several economic and environmental reasons [1-5].
Processes followed in the recycling of aluminum have a direct effect on the microstructure of the alloy, which explains its mechanical behavior. So, to understand the evolution of the mechanical properties of such alloy, it is imperative to carry out an exhaustive study of its microstructure affected by the implementation process parameters of on the one hand, and by the used metallurgical factors on the other hand.
Results of previous studies show the reliability of the application of pressure in improving the mechanical properties of different alloys [6-8]. Many researchers contend that the application of pressure on molten metal during solidification can have several effects such as changing the freezing point according to the Clausius-Clapeyron relationship [9], changing the cooling rate [10,11] and reducing porosity and shrinkage, which improves the microstructural properties of alloys. As a result, the mechanical proprieties were improved [12,13]. Many studies have correlated the mechanical properties and the cooling rate according to the Hall-Petch relation [14,15].
Others researches are focused in the improvement of Aluminum alloys proprieties; Teng Liu et al. have prepared Aluminum-aluminum bimetal by casting liquid A356 aluminum alloy onto 6101 aluminum extrusion bars and solidifying under applied pressure [16]. Patel et al. have made an attempt to predict the secondary dendrite arm spacing (SDAS) utilizing Mamdani, Takagi and Sugeno based fuzzy logic approaches. The performance all models are compared in making the prediction of SDAS in squeeze casting [17]. P. Senthil et al. [18] have also produce the AC2A aluminum alloy castings through direct squeeze casting process. Taguchi method and genetic algorithm were employed for process optimization in order to produce high quality castings. They have reported that castings obtained for optimum squeeze casting condition exhibited better grain refinement in microstructure and around 65 %

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Table I: Chemical composition of the investigated Al-Mg-Si alloy

Al	Si	Mg	Zn	Fe	Cu	Mn	Ni	Cr	Pb
98.8	0.55	0.20	<0.15	0.36	0.0149	0.0278	<0.1	0.0344	<0.03

improvement in tensile yield strength than gravity die casting condition.

Many have used the squeeze casting as a process for preparing composite alloys because of its high productivity and excellent formability [19-22]. E. Hajjari et al. [23] showed that the appropriate pressures for manufacturing carbon fiber reinforced aluminum composites, by squeeze casting process, in uncoated and Ni-coated condition are 50 and 30MPa, respectively. B. Lin et al. [24] have prepared Al-5.0wt% Cu-0.6 wt% Mn alloys with different Fe contents by gravity die casting and squeeze casting. The difference in microstructures and mechanical properties of the T5 heat-treated alloys was examined by tensile test, optical microscopy, deep etching technique, scanning electronmicroscope and electron probe micro-analyzer. They have shown that the squeeze cast alloys with different Fe contents have superior mechanical properties compared to the gravity die cast alloys, which is mainly attributed to the reduction of porosity and refinement of Fe-rich intermetallic and a (Al) dendrite observed in T5 heat-treated gravity die cast alloy. Souissi N. et al. investigated the relationship between the ultimate tensile strength, hardness and process variables in a squeeze casting 2017 a wrought aluminum alloy to, in a first study [25], and the Improvement of ductility of this alloy, in a second study, using the Taguchi method [26]. The objectives of the Taguchi method for the squeeze casting process are to establish the optimal combination of process parameters and to reduce the variation in quality between only a few experiments. The experimental results show that the squeeze pressure significantly affects the microstructure and the mechanical properties of 2017 A Al alloys [25].

In this work, we used squeeze casting for recycling an aluminum alloy; the effect of pressure variation on its metallographic structure and mechanical properties evolution were investigated. The pressures applied during solidification were 0, 50, 75, 100 and 150MPa. We were supposed to predict the effect of pressures on cooling rate changes and grain refinement; we were also supposed to find a compromise between microstructural results and mechanical behavior of the samples. The suitable heat treatment which could lead to better mechanical properties is also selected

2. Material and Methods

2.1 Material and Squeeze casting method

The material studied is an aluminum scrap. The method of X-ray fluorescence spectrometry was used to the determination and quantitation of the alloy. The chemical composition is shown in table I.

Squeeze casting is a technical process in which metal is solidified under pressure, and can be regarded as a combination of die-casting and closed die forging.

We used in this work for Squeeze casting experiments, a hydraulic press which is shown in figure 1.

The metal was melted in a crucible furnace at 750 °C. Before casting, these steps had to be followed to avoid thermal shocks in the mold during casting:

A mixture compounded of 50% graphite, 5% sodium silicate and 45% water was applied on the surface of the mold and the crucible furnace. The die was heated to a temperature of 250°C (pre-heating

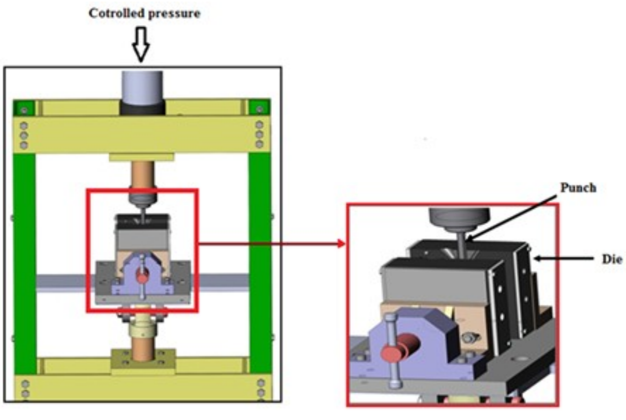

Fig.1. Experimental setup of squeeze casting process

Table II: Experimental conditions used for squeeze casting of the samples

Variable parameter	Pressure, P(MPa) P = 0, 50, 75, 100, 150
Fixed parameters	Melt temperature, Tm (◦C) Tm = 750 Die temperature, Td (◦C) Td = 250

temperature). This temperature was controlled by a thermocouple and temperature controller. Table II shows the experimental conditions used for the squeeze casting of the samples.
The melt was poured into the die. Pressure was applied immediately through the punch and held until solidification was achieved.

2.2 Metallographic examination
The microstructural observations were performed by an optical microscope and a scanning electron microscope (SEM). For optical microstructure observations, each specimen section was preliminarily subjected to a metallographic preparation etched with 1% HF aqueous solution to

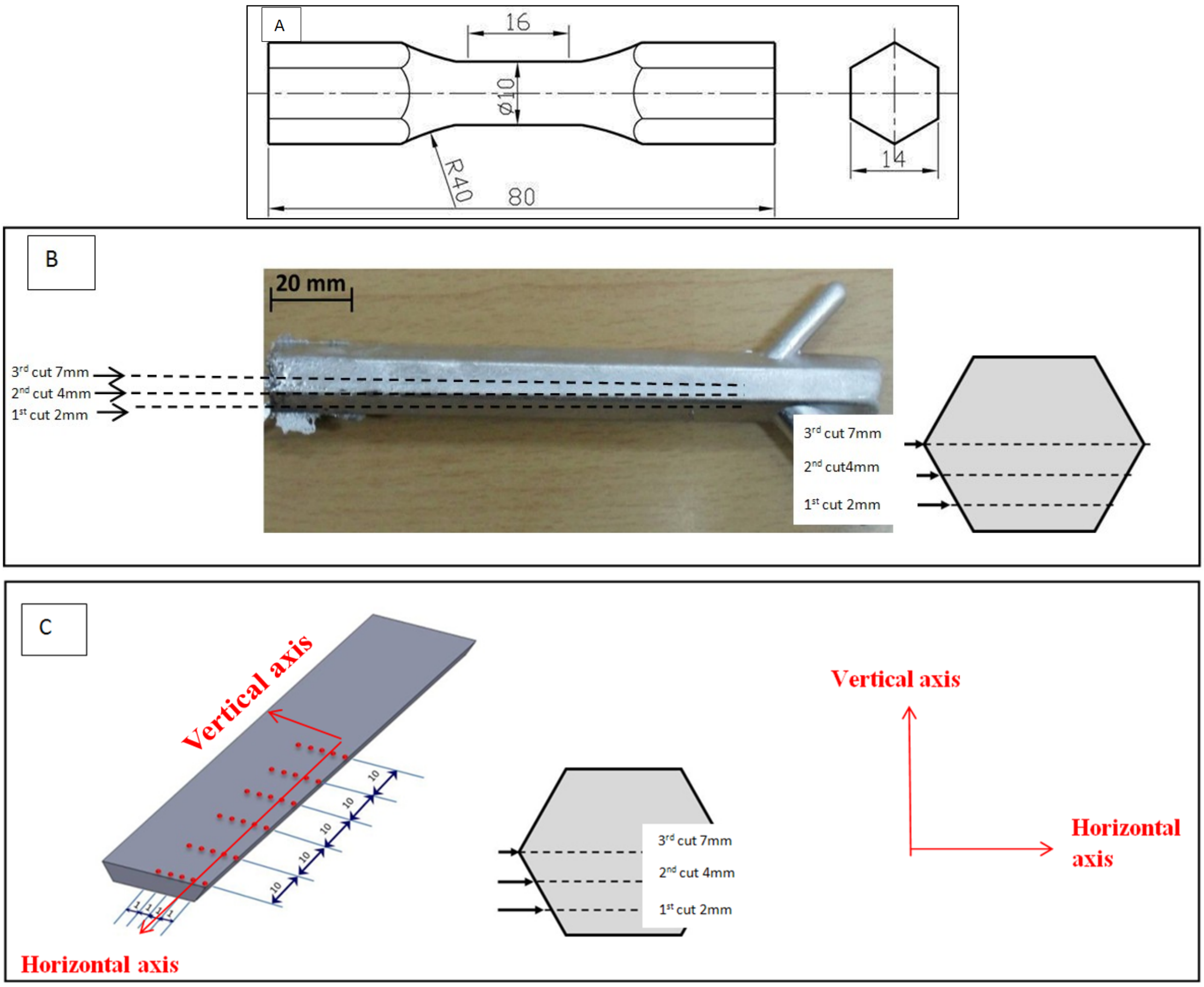

Fig.2. (A) Dimension of tensile test specimen (mm), (B); Schematic of the sample three cuts, (C) Schematic of different points sectionsof hardness tests

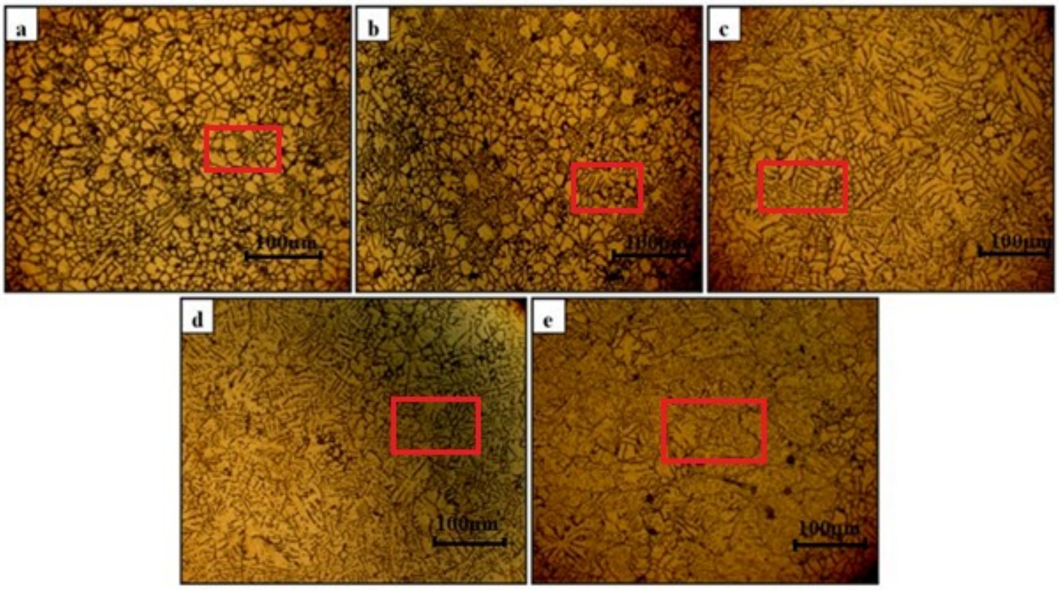

Fig.3. Microstructures of squeeze cast Al-Mg-Si alloy:
(a) 0MPa (gravity cast specimen), (b) 50MPa,(c)75MPa,(d)100MPa.

reveal the microstructure and observed with an optical microscope.

2.3 Tensile testing

In order to perform tensile tests, the samples were prepared by CNC lathe machine according to the standard (ISO 6892-1:2009) form. The tests were performed under displacement control with a strain rate start at 5mm/min. An extensometer (gage length of 14 mm) is attached with two rubber bands to the central part of the specimen. The geometry of the tensile specimens is shown in Figure 2(A).

2.4 Heat treatment analysis

There are structural hardening treatments involving three major stages: annealing, quenching and aging. In order to maximize the precipitation density and hardness of the material, the appropriate choice of aging time was selected after different hardness tests at 160°C after different aging time for 1, 2, 3, 4, 5, 6, 7 and 8 hours. Maximum of hardness was obtained after 6 hours. So, the samples were annealed for 6 hours at 500 ° C, quenching in water at atmospheric temperature and aging at 160 ° C for 6 hours.

The strength of Al-Mg-Si alloys is average after maturation, but it increases after aging [27].

2.5 Cooling rate analysis

The cooling rate is the most important parameter affecting casting microstructure evolution and mechanical properties. In order to study the effect of pressure on cooling rate, three longitudinal cuts were performed for 3specimens solidified under 0,100 and 150MPa external applied pressures, by a robot machine at a distance of 2, 4 and 7 millimeters from the surface area to the sample radius. Fig.2 (B) shows the three sample cuts.

2.6 Hardness testing

The hardness Vickers tests were performed by a MEKTON Vickers Hardness Tester, on different points sections along two axes of the sample, shown in fig.2(C):

- The horizontal axis (graduated 10 mm, 20 mm, 30 mm, 40 mm and 50 mm)
- The vertical axis (graduated in 1 mm, 2 mm, 3 mm, 4 mm, 5 mm).

3. Results and discussion

3.1 Microstructural characteristics

A comparison between different microstructure samples aspects, solidified under 0, 50, 75, 100 and 150MPa external applied pressures, is provided in Fig. 3 and its higher magnification are shown in Fig. 4.

All the microstructures exhibited distinct regions of fine and coarse dendritic structure. For the gravity casting sample (0MPa), fig 4(a) shows that the microstructures are formed by larges grains. With increasing pressure, the volume fraction of fine structure increased however, the coarse dendritic structure disappeared progressively. It

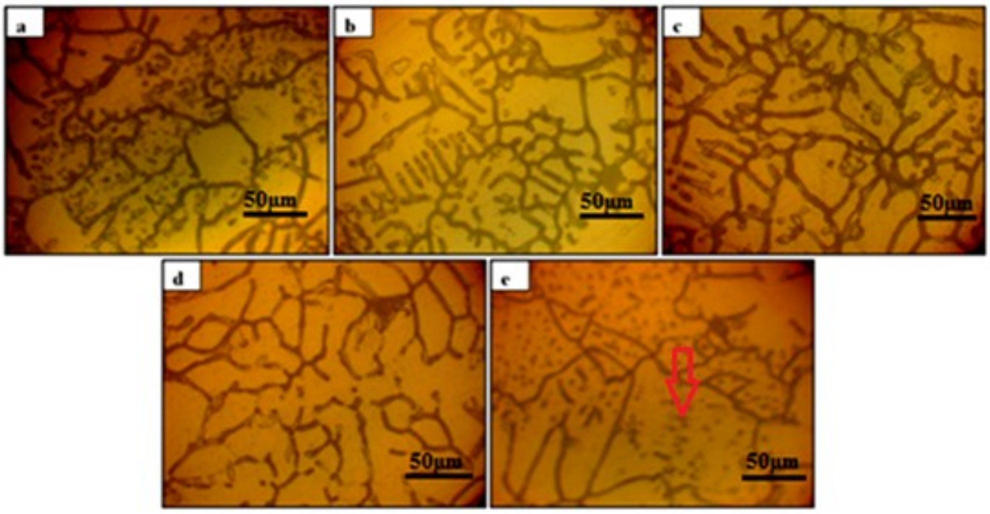

Fig. 4. Microstructures of squeeze cast Al-Mg-Si alloy: (a) 0MPa (gravity cast specimen), (b) 50MPa, (c) 75MPa, (d) 100MPa.(Magnification×500)

was reported that the melting point (liquidus temperature) of most metals and alloys increases under pressure according to Clausius-Clapeyron equation [9]. The surface aspect is clearly dependent on pressure due to the better metal-mold matching during squeeze casting. Obviously, the increase of undercooling degree and heat-transfer coefficient will result in the refinement of the grain size of squeeze casting alloy. At a 100MPa external applied pressure, the grains size became more homogenous and finer. The grain boundaries also seem finer (Fig. 3 (d) and Fig. 4 (d)). However, with application of 150MPa external pressure, the grains size was noted to undergo sharp growth; it can be observed that acriquability and a destruction phenomena of the sample are begin to appear. The microstructure of the alloy is influenced by the cooling rate evolution during casting. Gravity casting (0MPa) results in the formation of a heterogeneous microstructure: containing three different areas (skin area, “basaltic” area and “equiaxed” area) [28]. Applying pressure allows homogeneous micro-structure and grain size.

Table III Different sections of the specimens

A-a	first cut, in the middle of the specimen
B-b	second cut, in the middle of the specimen
C-c	third cut in the middle of the specimen
A-d	first cut, at the edge of the specimen
B-e	second cut, at the edge of the specimen
C-f	third cut, at the edge of the specimen

G.S. Reddy and al. showed that, when solidified under pressure, the alloy systems exhibited distinct regions of fine and coarse dendritic structure (Bi-modal dendritic structure). Hence, the variation of the cooling rate at different points in the mold caused the formation of a bi- modal structure. [29]

The various longitudinal cuts carried on 3 samples under 0MPa, 100MPa and 150MPa pressure, in order to study the effect of pressure on cooling rate, are given in Table III and the micrograph results of the samples are shown in Fig5.

There are changes in the microstructure due to the cooling rate evolution. At 0MPa (Fig. 5-I (Bb)), dendrites appear in the middle of sections with no growth preferred direction; they grow in the heat flow direction. The grains are thinner in the sample edge due to heat transfer improvement on the mold walls. The micro-pores that appear in the middle of the sample decrease the mechanical properties.

During squeeze casting, with increasing applied pressure, the microstructure tends to be homogeneous. At 100MPa, the grains sizes decrease and the grains boundaries density increases due to intense germination and fast cooling (Fig. 5-II). As a result, the micro-porosity becomes small and few. It is well established that the application of an external pressure during the solidification of the metal activates the different

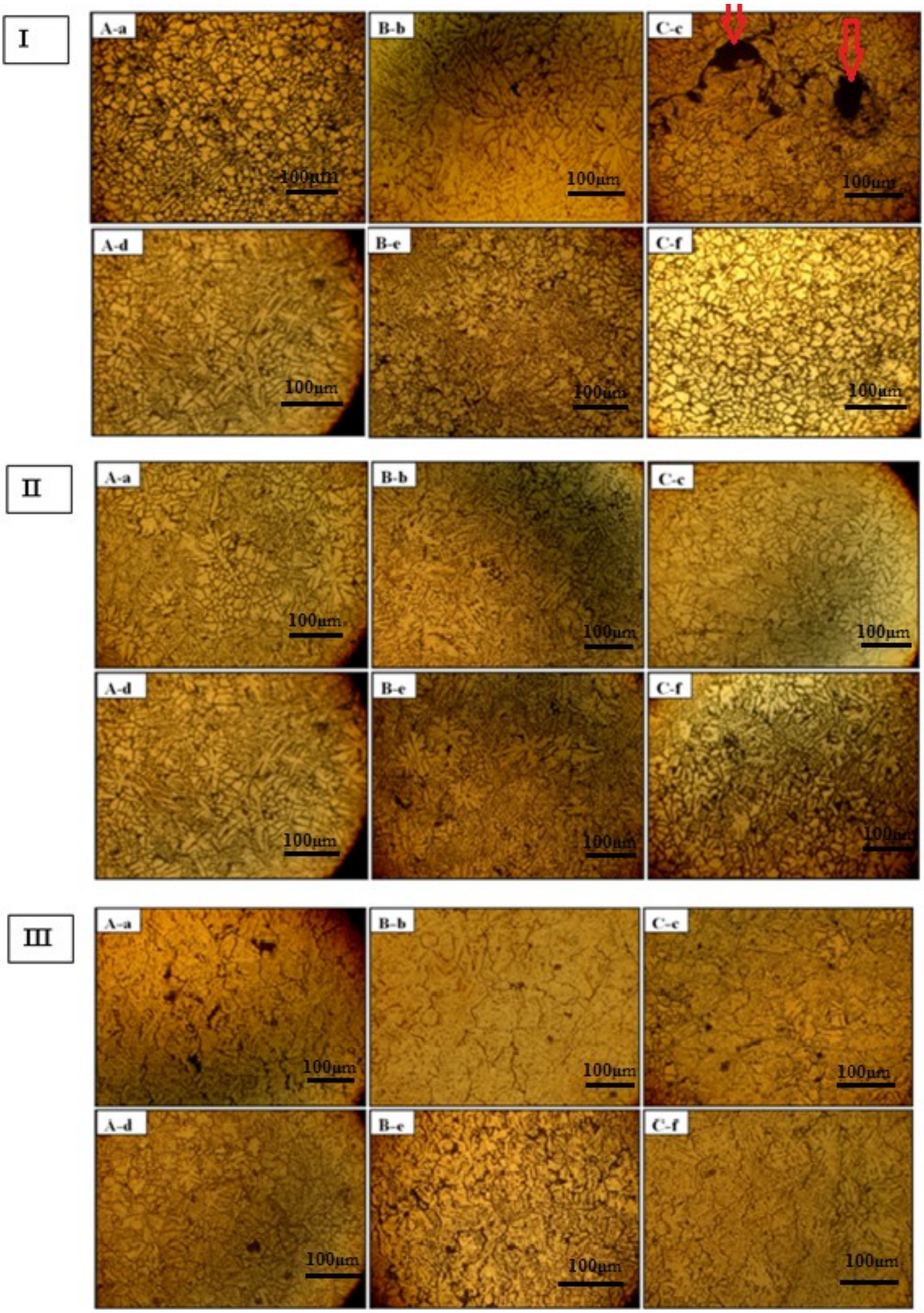

Fig. 5. Optical Micrographs of metallographic sections of as- cast alloy I: 0MPa, II: 100MPa squeezed sample, III:150MPa squeezed sample

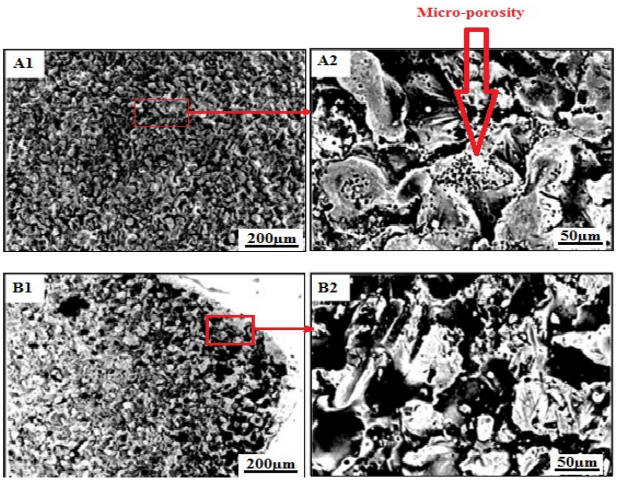

Fig. 6. SEM micrographs of the microstructure evolution of as-cast alloy at (0MPa) squeezing pressure: **(A1, A2)** in the middle and (**B1, B2**) at the edge of the specimen.

feeding mechanisms and hinders the shrinkage porosity formation. With increasing external pressure until 150MPa, the grains size increased intensively as a result of their coalescence (Fig. 5.III).

We have performed a comparison between different microstructure aspects for 0MPa, 100MPa and 150MPa applied pressures, in two different points (the center and the edge) with different magnifications.

The Observations by scanning electron microscope also revealed the evolution of microstructures morphology and the defects size formed during solidification according to pressure. At 0MPa, Figs. 6(A1, A2) show that micro- porosity have large size, in the middle of the specimen. This micro- porosity results from the exhaust gas during metal casting. In squeeze casting, the applied pressure can help the liquid metal to fill in the micro-pores. In this study, it was found that micro-porosity was eliminated completely when the pressure reached 100MPa(Fig. 7(A1, A2)). Therefore, we conclude that at atmospheric pressure (0MPa), the liquid metal was cooled simultaneously from the sample wall to its middle; defects such as microporosity and shrinkage gas were blocked at the center. With increasing-pressure (100MPa) they accumulated and migrated at the edge then released at specimens surface. But at 150MPa they reappeared in small size on the sample edge (Fig. 8(B2)).

Precipitates were observed in all the micrographs and localized with high concentration at the specimen edge at 100MPa. (Figs. 7(B2)).

3.2. Mechanical characteristics

To characterize the mechanical behavior of the metal, tensile test was performed on specimens in rough castings. Elasticity (Rp 0.2), Rm, elongation and Young's modulus of the investigated alloy at different squeeze casting pressures are shown in Fig.9.

It is noted that the applied pressure under 100MPa had a significant effect on the tensile properties of the alloy; these parameters increased with increasing pressure. The values of $Rp_{0.2}$, Rm and the elongation of the alloy squeezed cast at

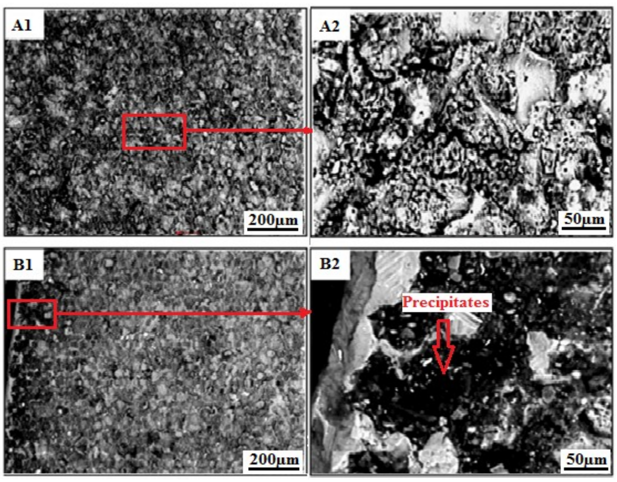

Fig. 7. SEM micrographs of the microstructure evolution of as-cast alloy at (100MPa) squeezing pressure: (**A1, A2**) in the middle and (**B1, B2**) at the edge of the specimen.

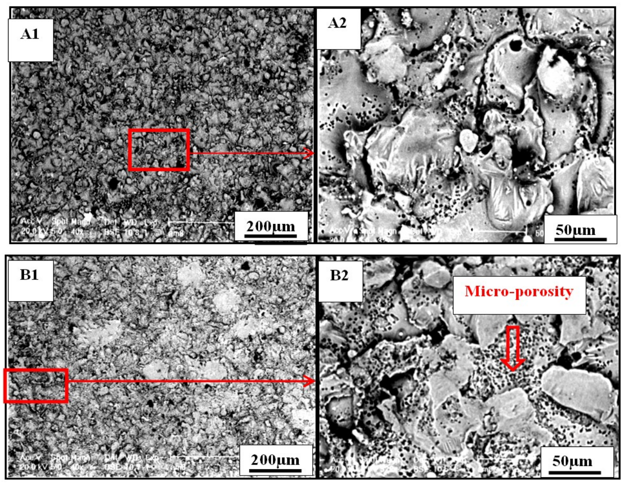

Fig. 8. SEM micrographs of the microstructure evolution of as-cast alloy at (150MPa) squeezing pressure: (**A1, A2**) in the middle and (**B1, B2**) at the edge of the specimen.

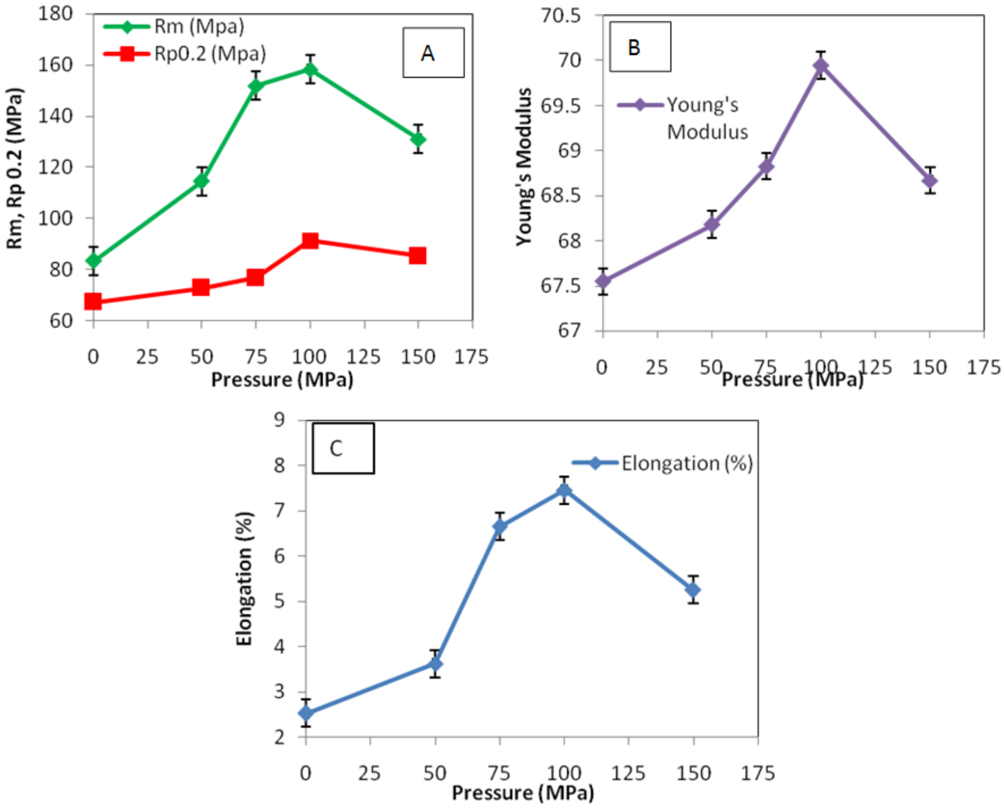

Fig. 9. Curves of (A): Rp0, 2 (MPa) and Rm (MPa), (B) Young's modulus (GPa) and (C): elongation (%) of squeeze cast specimens at different external applied pressure.

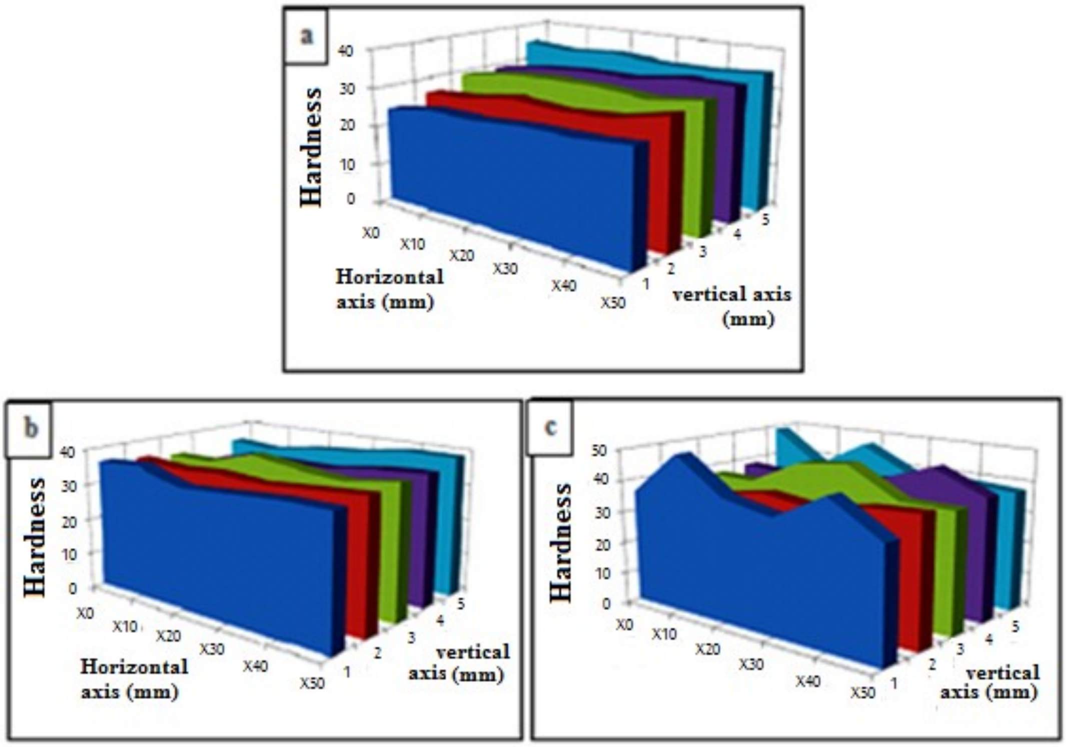

Fig. 10. The microhardness measurements of thee three specimens from the edge to the middle of (**a**): 0MPa, (**b**): 100MPa squeezed sample, (**c**): 150MPa squeezed sample.

100MPa were 91.4MPa, 158.27MPa and 9.46%, respectively. The increasing of external pressure until 150MPa caused the embrittlement and damage of the specimen. This result can be explained by this phenomenon: The strain during squeeze casting creates a high density of dislocations and other defects. Since the hardness of the metal is a function of the dislocations propagation, this later was hindered by obstacles such as grain boundaries, precipitates and other dislocations. With increasing of external pressure until 150MPa, density of dislocations increases intensively and its movement was totally hindered, as a result we can note a sharp increase of the hardness, causing the fragility of the alloy.

The hardness measurements of the three specimens are shown in Fig.10.

It is shown that hardness increases with pressure. At 0MPa, (fig.10(a)) the hardness measurements increases from the sample middle to the edge and we note the existence of a significant slope. With increasing pressure, hardness measurements are more homogenous. At 100MPa, the hardness values are 37HV and 38HV in the middle, and 39HV at the edge (fig.10(b)), therefore the histograms surfaces tend to be horizontal. This explains the mechanical properties homogeneity of the specimen. At 150MPa, hardness increases intensively and randomly with no preferred direction (fig.10(c)). The material heterogeneity is caused by the anomaly observed in sample microstructures; Microhardness measurements results are well justified by the micrographic results.

CONCLUSIONS

- A compromise between the microstructural and mechanical experimental results is found as a result of the application of pressure during casting of the alloy.
- The external pressure P=100 MPa results in optimal microstructural characteristics; the increase of undercooling degree and heat-transfer coefficient will result in the refinement of the grain size and the reduction of micro-porosity. So, the improvement of the mechanical properties of squeeze casting alloys
- At 150MPa external applied pressure, an anomaly results. The grain growth and high concentration of linear and intrinsic defects in the alloy cause the damage of the specimen.

Acknowledgements: Authors would like to thank Leila Mahfoudhi from the English Language Unit at the Faculty of Sciences of Sfax (Tunisia) for accepting to proofread and polish the language of this paper.

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