

# Mechanical properties and microstructural evaluation of AA5013 aluminum alloy treated in the semi-solid state by SIMA process

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## Abstract

The microstructure and mechanical properties of AA5013 aluminum alloys prepared by strain-induced melt activation (SIMA) process were studied to investigate the effects of cold working and heat treatment conditions. The specimens subjected to deformation ratios of 30 and 50% and various heat treatment time and temperature regimes were characterized in the present study. The results revealed that for the desired microstructure of the alloy, the optimum heat treatment temperature and time were 650 °C and 60 min, respectively, for both deformation ratios. However, the specimens with 50% cold working exhibited more brittle behavior while they contain finer grains with uniform distribution along the cross-sections as compared to 30% cold working.

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## 1. Introduction

Semi-solid metal processing (SSF) technology, developed at MIT during the 1970s by Spencer and co-workers, has received considerable interest in recent years [1]. In this technique, the alloy is heated to temperatures at which solid and liquid phase coexist in equilibrium and is then subjected to a forming process. The SSF technique offers some advantages, such as reduced flow stress during shearing, over the traditional metal forming methods, namely casting, forging, and powder metallurgy [2]. However, the technique requires alloys with a fine equiaxed, dendrite-free, grain structure prior to formation of the semi-solid state.

A number of methods, such as mechanical or electromagnetic stirring, the addition of grain refining elements, spray casting, and rapid cooling, have been reported to obtain near equiaxed grain structures. Alternatively, the strain-induced melt activation (SIMA) process produces the desired structures by deformation and a following heat treatment in the mushy zone. Parameters such as heating time and temperature, and the degree of cold working, are critical factors

in controlling the semi-solid microstructures in the SIMA process [1–4]. It has been shown that the microstructure of an alloy prepared in the semi-solid state depends on its microstructure prior to partial remelting, so it is important to study the initial microstructure and subsequent evolution process during partial melting.

The most common alloys used for the SSF technique have been 7xxx or 2xxx series wrought Al alloys or certain cast alloys [3–9]. To our knowledge there is no study reported in the literature about the semi-solid behavior of AA5013 Al alloys. The 5xxx alloys have found a large variety of applications including architectural, ornamental, decorative trim, cans and can ends, household appliances, streetlight standards, marine craft, cryogenic tanks, crane components, and automotive structures [10].

The 5xxx series alloys, in which the major alloying element is magnesium, exhibit moderate-to-high-strength work-hardenable characteristics. Alloys in this series possess good welding characteristics and good resistance to corrosion in marine environments. However, certain limitations need to be placed on the amount of cold work and the safe operating temperatures permissible for the higher-magnesium alloys to avoid susceptibility to stress-corrosion cracking.

The present study investigates the effects of heat treatment time, temperature, and the degree of cold working on the

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microstructure, i.e. grain globularization of SIMA-processed AA5013 alloys.

## 2. Experimental

The AA5013 Al alloy was procured in the tempered condition as a solid plate and its chemical composition, determined using an ARL Fisons model 3460 metal analyzer system, is listed in Table 1. A 100 t hydraulic press was used for cold working of the samples. The specimens were compacted in height by either 30 or 50% of the original. The deformed samples were then heated to various temperatures within the range of the liquidus–solidus zone and maintained at those temperatures for various holding times in the range 20–60 min as listed in Table 2. The microstructure of the materials after heating in the semi-solid temperature range was examined by optical microscopy. The compositional variations occurring due to diffusion of solute elements in the semi-solid transition were investigated by SEM–EDX analysis. Rockwell B indentation experiments were performed to determine the effects of heat treatment conditions on the mechanical properties of cold-worked AA5013 alloys.

Table 1  
Chemical composition of the AA5013 Al alloy (as-received in the tempered condition)

	Composition (%)
Si	0.228
Ti	0.012
Fe	0.266
Cr	0.001
Cu	0.045
Ni	0.0032
Mn	0.35
Pb	0.0012
Mg	3.35
Sn	0.0021
Zn	0.008
Al	95.72

Table 2  
The heat treatment conditions of cold-worked alloys for SIMA process

Temperature (°C)	Time (min)
575	40
575	50
600	20
600	30
600	40
600	50
600	60
625	40
650	30
650	45
650	60

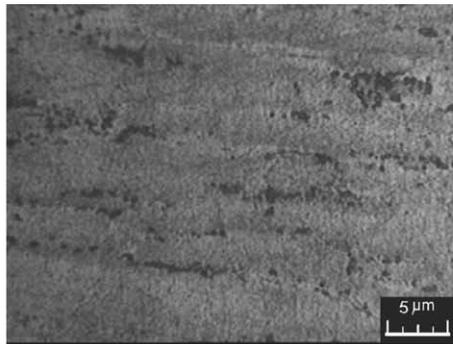
## 3. Results and discussion

The microstructures of the cold-worked (at 30 and 50% height reduction) and heat-treated AA5013 alloys were examined to determine the optimum treatment conditions for the formation of the desired near-equiaxed spherical grains. In Fig. 1, the optical micrographs show the typical microstructures of the alloys heat-treated under various conditions after 30 and 50% cold working. For both ratios, cold-worked structures with elongated grains were obtained and those structures were still observed in specimens heat-treated up to 600 °C for 60 min.

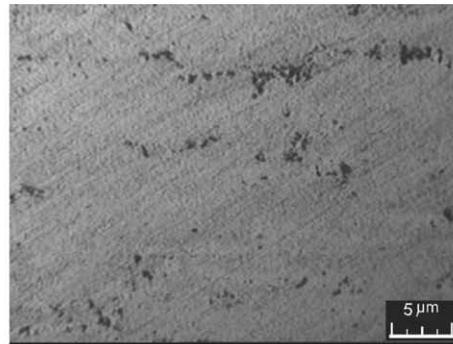
It was found that the recrystallization of the cold-worked structure initiated at about 625 °C after 70 min. On the other hand, recrystallization was completed when the cold-worked alloy was treated at 650 °C for 45 min. However, under this condition, sharp-edged grains formed. Ideally, rounded solid grains, without any entrapped liquid phases, are necessary to obtain the highest fluidity in the SSF process [11]. As shown in Fig. 1, the microstructure of the sample treated at 650 °C for 60 min shows grains with an almost equiaxed form. At higher temperatures or longer holding times, it was observed that the average diameter of the grains increased and that their shapes tended to be rounded with increasing liquid fraction. It is known that such a structure results in a higher fluidity, and also that the formation of porosity in the final structure degrades the mechanical properties of the material.

It was also found that the degree of cold working has some effect on the grain size variations along the cross-section of the alloy. For 50% cold working, it was found that the grains were of almost uniform shape throughout the sample cross-section, and grain sizes were finer compared to the 30% cold-worked alloy. On the other hand, it was found that for 30% cold working, the grain sizes exhibit less uniformity from the outer surface to the center of the sample as illustrated in Fig. 2. Note that the micrographs in Fig. 1 for the 30% cold-worked samples were taken from regions representative of typical grain sizes. It is known that non-uniformity of the microstructure may reduce the effectiveness of the SSF process.

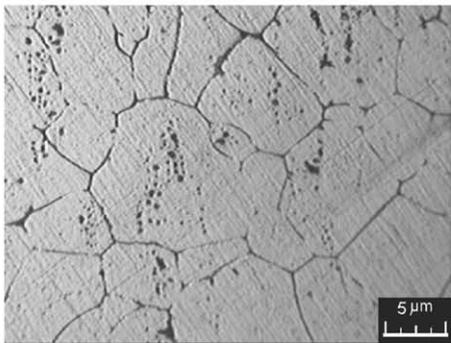
Fig. 3 shows the effect of cold working and heat treatment conditions on the hardness of the alloy. As expected, the hardness values of the specimens deformed to 50% were higher than those for the specimens deformed to 30%. However, as the heat treatment time or temperature increased, the differences between the hardness values of the 50 and 30% cold-worked material became almost insignificant. This may be associated with the progress of recrystallization and reduction of lattice strains or residual stresses with increasing time and temperature. Also, the hardness values were observed to increase as the holding time was increased at a given temperature. This may be due to solid solution hardening of AA5013 alloy by secondary solid elements at elevated holding time and temperature. In addition, the specimens with 50% cold working exhibited more brittleness than those with 30% cold working.



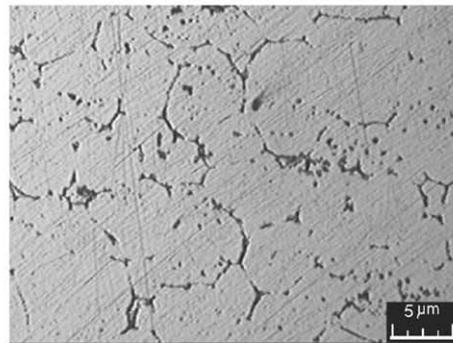
30% def. and heat treated at 600 °C - 60 min.



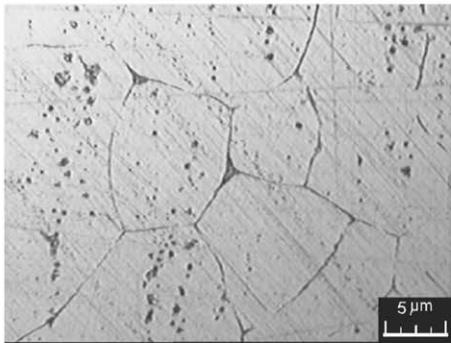
50% def. and heat treated at 600 °C - 60 min.



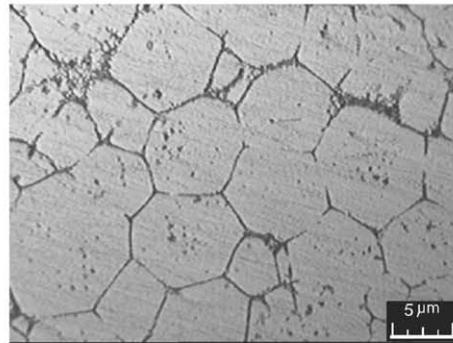
30% def. and heat treated at 650 °C - 30 min



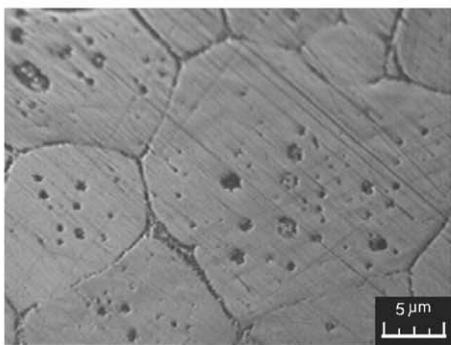
50% def. and heat treated at 650 °C - 30 min



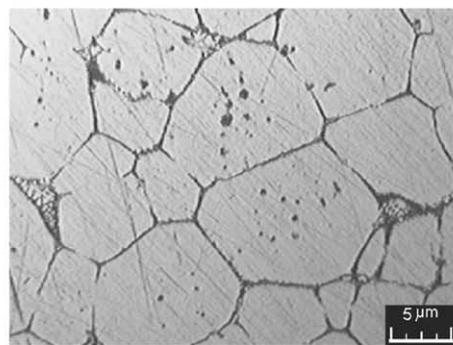
30% def. and heat treated at 650 °C - 45 min



50% def. and heat treated at 650 °C - 45 min



30% def. and heat treated at 650 °C - 60 min



50% def. and heat treated at 650 °C - 60 min

Fig. 1. Optical microscopy of AA5013 after cold working at 30 and 50% and heat-treated at various times and temperatures.

The compositional variations due to the microsegregation of solute elements from grain center to grain boundary were examined by SEM–EDX. Fig. 4 is an SEM micrograph showing the grain structure of the Al alloy examined

with SEM–EDX. Typical examples of those variations for 30 and 50% cold work are listed in Table 3. The results indicated that concentrations of Mg, Si, O and other alloying elements increased significantly at the grain boundaries

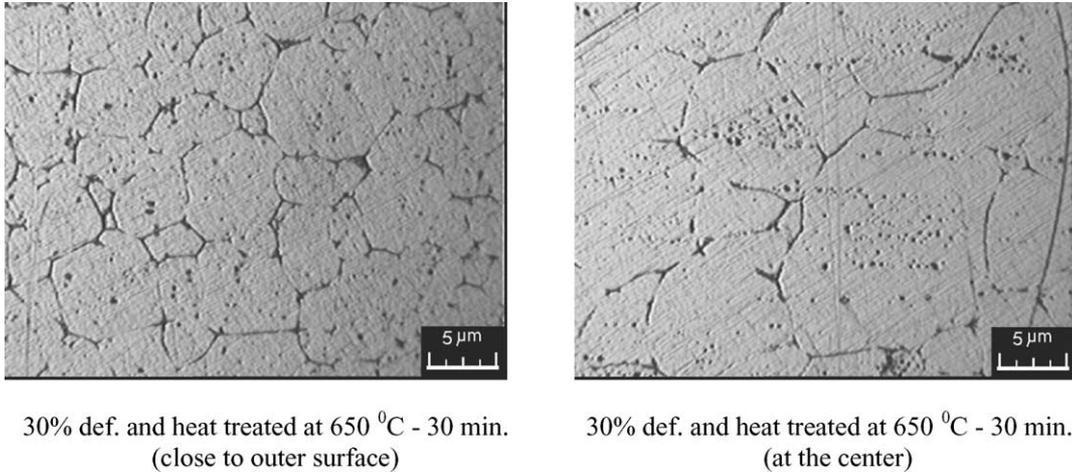


Fig. 2. Optical micrographs showing the change of grain size along the cross-section from outer surface to the center for the 30% deformed sample.

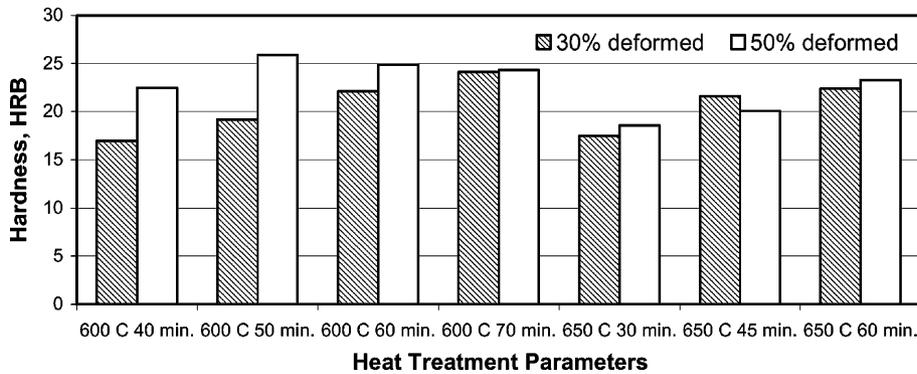


Fig. 3. Results from hardness measurement of AA5013 after various process conditions.

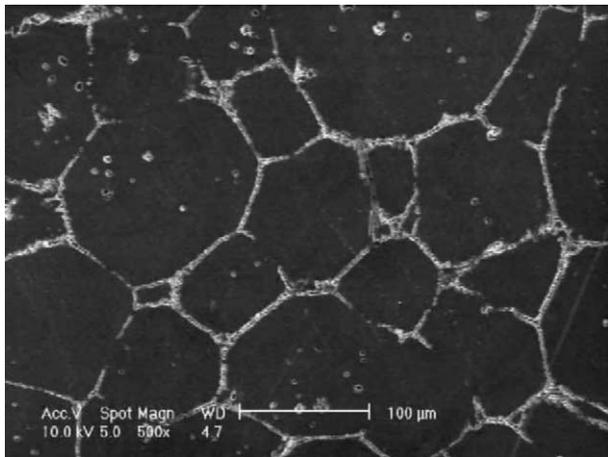


Fig. 4. SEM micrograph showing microstructure of AA5013 alloy (after 30% cold deformation and heat-treated at 650 °C for 60 min) examined with SEM-EDX.

Table 3

SEM-EDX analysis results for AA5013 after semi-solid process with 30 and 50% cold working and heat-treated at 650 °C for 60 min

Grain center			Grain boundary		
Element	wt.%	at.%	Element	wt.%	at.%
<b>30% cold-worked</b>					
O	1.95	3.29	O	1.99	3.44
Mg	4.60	5.10	Mg	7.15	8.13
Al	90.46	90.24	Al	84.14	86.18
Si	0.39	0.38	Si	0.94	0.92
Ag	0.47	0.12	Ag	2.00	0.51
Ti	0.41	0.23	Ti	0.71	0.41
Fe	1.21	0.58	Fe	0.00	0.00
Pb	0.39	0.05	Pb	2.24	0.30
Bi	0.11	0.01	Bi	0.83	0.11
<b>50% cold-worked</b>					
O	0.94	1.60	O	1.66	2.84
Mg	2.83	3.18	Mg	8.23	9.24
Al	92.82	93.96	Al	83.05	83.97
Si	0.00	0.00	Si	2.10	2.04
Ag	1.87	0.47	Ag	2.06	0.52
Ti	0.36	0.21	Ti	0.23	0.13
Fe	1.18	0.57	Fe	0.36	0.18
			Ni	1.95	0.91
			Mn	0.35	0.17

and also around the inclusions within the grains. The oxygen content is expected to be responsible for the extent of porosity formation that may arise at higher temperatures or process times.

#### 4. Conclusions

The SIMA process is an effective method for producing alloys with the spherical grains required for SFF treatment. The SIMA process is appropriate for AA5013 aluminum alloys but with certain limitations. As the heat treatment temperature and/or the holding time is increased, equiaxed-form grains can be obtained. On the other hand, the grain size tends to increase with increasing time and temperature. The equiaxed grains may increase the fluidity during the SFF process, while the elevated oxygen content may degrade the mechanical properties due to porosity formation. Also, it was observed that grains with smaller diameter and higher uniformity along the cross-section of the specimens can be obtained with 50% cold working, but this condition exhibited greater brittleness than for 30% cold working. Based on the observations in this study, it may be concluded that the optimum condition for semi-solid forging of AA5013 alloy is about 650 °C for 60 min.

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