



Effect of deep sub-zero treatment on mechanical properties of AA5XXX aluminum plates adjoined by MIG welding technique

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Abstract. In this study, mechanical and microstructural characteristics of AA5XXX aluminum plates connected by MIG welding were investigated. Thus, after several preliminary experiments done to ensure quality requirements of the parameters, specimens in the form of long aluminum plates were welded, and then some of them were spared for the subsequent sub-zero treatment at about 146°C for 12 and 24 h, respectively. To investigate the effect of this super cooling on the welded parts, several tensile, bending, and hardness tests in conjunction with micro-structure examinations were carried out and results were discussed in detail. It was concluded that during sub-zero treatment, micro-structure gets finer and stabilized, positively affecting the overall strength of the material. There is no significant difference in the overall strength of the material right after the sub-zero treatment and that of the specimens rested for one year at room temperature after cryogenics treatments.

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1. Introduction

Aluminum and its alloys are commonly used in various industrial and structural engineering applications due to their low density and high strength-to-weight ratio. Various types of Al-alloys were extensively studied in terms of joining [1-4]. Aluminum is one of the most abundant elements on the earth's crust, started to be used commonly at the end of the 19th century in engineering applications. Currently, aluminum alloys are

used in high-tech fields such as electronics, automobile, transportation, sporting goods, and aerospace industries [5]. Today, value and usage of these materials are effectively classified according to their possibility of welding and joining capability and giving long service lives [6-10].

Cryogenic treatment is a supplementary process for conventional heat treatment. Unlike the outcomes of conventional surface treatments, it is an inexpensive treatment that affects both the core structure and properties of the components. More than three decades, studies on cryogenic impacts, particularly for the behavior of tool steels have been done seriously. But so far, very little knowledge exists on how cryogenics affects non-ferrous materials such as aluminum. However, literature survey commonly reveals

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that mechanical strength of materials mostly increases significantly after being submitted to several cycles of subzero treatments [11–14].

Aluminum Alloy (AA) 5083 is the marine-grade alloy used in the construction of passenger, freight, and naval containers. Owing to the demanding nature of the service conditions and environments in which marine vessels operate, welded components and structures are susceptible to high level of loads, which may cause micro-structural degrade and final catastrophic failure. As a necessity of using tremendous amount of aluminum alloys in ship construction industry, a great deal of research has been carried out focusing on the long-term performance of the welded joints [15–17]. There are only a few papers examining the microstructural changes that result in the failure of the joint which is mostly introduced during the process of welding [18]. AA5083 alloy is preferred in many applications due to its acceptable grade of weldability, strength, and high corrosion resistance. However, welding of such alloys after severe strain hardening mostly results in substantial reduction of strength in the Heat Affected Zone (HAZ) [19]. Decrease in strength is unavoidable, but special care should be taken to avoid welding defects that may further reduce the overall strength of the material. Thus, possible defects have to be inspected routinely in the vicinity of the weldment, repairing the weldment can be usually employed to extend service life of the component. Despite wide application of the present alloy and interest in improving its properties by additional alloying, there is still a lack of detailed mechanical characterization of this material and study of the effect of subsequent cryogenic treatment on its microstructure. Thus, in this study, the MIG welded samples of AA5083 alloy were soaked at -146°C for 12 h and 24 h, and then rested at room temperature for a while before investigating the microstructure, hardness, bending, and tensile properties of the metal products.

On the other hand, after the cryogenic treatments, some samples were kept at room temperature on purpose for about a year to check the stability of

the mechanical strength and how the material stands for such a long time.

2. Experimental work and materials

2.1. Materials

AA5083 aluminum alloy was chosen for studying the effect of cryogenic treatment. The chemical composition of the samples ($5 \times 500 \times 1000 \text{ mm}^3$ in dimension) analyzed by a vacuum spectrometer is presented in Table 1. AA5336 (AlMg5Cr) wire as a filler material was used for the joining processes, where chemical composition of the filler is also given in Table 1. On the other hand, tensile properties of these materials are listed in Table 2.

2.2. Sample preparation and experimental details

Pre-welding cleaning procedures (oxide, stain, oil, and dirt removing) along with the steps taken to protect the molten weld zone were carried out to avoid contamination of the weld. A schematic diagram of the welded test plate and its welding consequence is shown in Figure 1. The plate was 5 mm thick, and therefore double-welding passes were deposited. The welds were produced using the ESABTM 400 type of semi-automatic welding machine. All the samples were obtained in the same orientation from flat position. During welding all parameters such as welding voltage and current, wire speeds, gas flow, and heat inputs

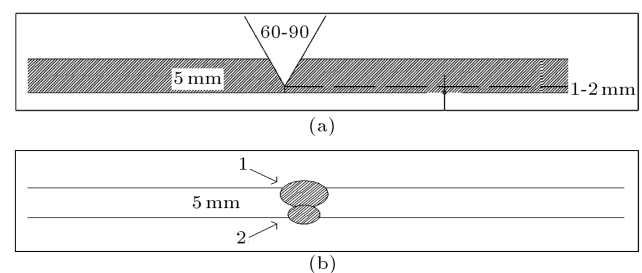


Figure 1. Schematic illustration of (a) groove and (b) welding sequence ($60-90^{\circ}$).

Table 1. Chemical composition of base and filler materials.

Materials	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Al
AA5083	0.12	0.243	0.073	0.489	4.85	0.108	0.075	0.035	Bal.
AA5356	5	0.4	0.1	0.5	0.1	0.1	0.1	0.15	Bal.

Table 2. Tensile properties of the materials.

Materials	Yield strength ($\sigma_{0.2}$), MPa	Ultimate tensile strength (σ_{UTS}), MPa	Elongation, %
AA5083	220	305	22
AA5356	250	310	23

Table 3. Details of the welding process variables.

Pass	Wire diameter (mm)	Current (A)/ voltage (V)	Gas flow (lt/min)	Torch angle (°)	Wire speed (mm/min)	Heat input (k_j/mm)
1	1.2	155-170/22.8-23.5	15	75-85	450-550	0.31-0.43
2	1.2	155-170/22.8-23.5	15	75-85	450-550	0.31-0.43

were measured. The welding machine characteristics and other parameters are given in Table 3.

Argon with a high rate of purity (%99.9) was used as a shielding gas during welding to prevent absorption of oxygen, hydrogen, and nitrogen from the atmosphere. Arc gap between the work piece and torch is 10-20 mm and the temperature between the passes is 120°C. Finally, cryogenic treatment was carried out in a nitrogen cryo-bath (-146°C) where the samples were soaked at that temperature for 12 h and 24 h. This regime of treatment is mostly preferred in the industry and most data available in the literature are listed for about this deep cryogenic level.

Samples were prepared according to EN 288-4 (TS EN ISO 15641-2) welding standards. Cutting process was made by water jet to avoid the interaction between heat and microstructure. Tensile test (TS EN ISO 4136) and bending tests (TS EN ISO 5173) were performed at room temperature and six samples were used for each condition. An average of these tests was presented in this study. Tensile tests were carried out by Instron MLF-type device of 60 kN capacity at a speed of 0.5 mm/min.

2.3. Metallography

$5 \times 25 \times 75 \text{ mm}^3$ samples were sectioned transversely into the weld direction for microstructural characterization and hardness measurements. A metallographic technique (ASTM E3-01 standard) was employed in grinding with various grades of SiC papers (i.e., 320-1200 meshes) followed by polishing using $3 \mu\text{m}$ and

$1 \mu\text{m}$ diamond grits. Samples were etched in the Keller's modified solution. The microstructure of samples was investigated by Nikon Eclipse LV 150 optical microscopy.

2.4. Hardness

Vickers micro-hardness measurements were taken from the center of the weld metal towards left and right sides by passing through HAZ to the base metal in the depth of 2 mm from the welding surface by using Instron micro-hardness tester with a load of 500 g.

3. Results and discussion

3.1. Tensile test results

Test results of Un-Treated (UT), 12 h Cryogenically Treated (12 h CT), and 24 h Cryogenically Treated (24 h CT) AA5083 samples are given in Table 4a, respectively.

As seen in Table 4, the yield strength, UTS, and elongation of untreated sample are lower than those of 12 h CT and 24 h CT samples. Compared to UT samples, yield strength of CT piece increases in 12%, ultimate tensile strength by 14%, and elongation by 2%, respectively. This can simply be attributed to the formation of ultra-fine microstructure and perfect penetration of the weldments. Similarly, it is obvious in Table 4b that UTS and elongation of UT are lower than those of 12 h CT samples. According to the results, compared to UT samples, CT pieces have an increase in yield strength by 12%, ultimate tensile strength by 14%, and elongation by 2%, respectively.

Table 4a. Tensile test results right after welding and cryonics process.

Sample	Yield strength ($\sigma_{\%0.2}$), MPa	Ultimate tensile strength (σ_{UTS}), MPa	Elongation, %
UT	200.12	297.1	21.12
12 h CT	264.13	296.22	23.85
24 h CT	294.44	392.24	23.99

Table 4b. Tensile test results one year later after welding and cryonics process.

Sample	Yield strength ($\sigma_{\%0.2}$), MPa	Ultimate tensile strength (σ_{UTS}), MPa	Elongation, %
UT	201.999	300.115	21.938
12 h CT	261.557	351.671	24.062
24 h CT	290.405	374.248	24.963

Alternatively, the results obtained right after welding and cryogenics process give lower mechanical values at least 3% compared to the samples rested for one year after welding and sub-zero treatment. However, in this study, as a whole, maximum change between all these is about 10%.

Similar to the above findings, several studies on the cryogenic evolution of welded samples claim that minor increases in both tensile strength and hardness are generally noticeable in weldments, particularly in the parent metal, such that after deep cryogenic treatment without sacrificing tensile strength, residual stresses mostly entrapped around the weldment can be released [20–23].

3.2. Bending test results

Typical photographs of bended samples after welding, such as UT, 12 h CT, and 24 h CT, are all presented in Figure 2. Because all the samples produced in this work are crack-free, bending characteristics can be mainly related to ductility of the material. Aluminum alloys are known as ductile materials. As can be noticed from Table 4, elongation values of welded samples are very similar.

3.3. Microstructure of joints

Typical macrostructures of the samples after welding, e.g. UT, 12 h CT, and 24 h CT, are given in Figure 3, where microstructural details are particularly shown in Figures 4–6 for different regions. According to macro-examination, perfect bonding and sufficient penetration are obtained. However, porosities can be traced out at the end of the root (Figure 3(c)). Porosity is a general term describing cavities or pores caused by gas and/or nonmetallic material entrapment in molten metal during solidification. There are many causes which include contamination, inadequate

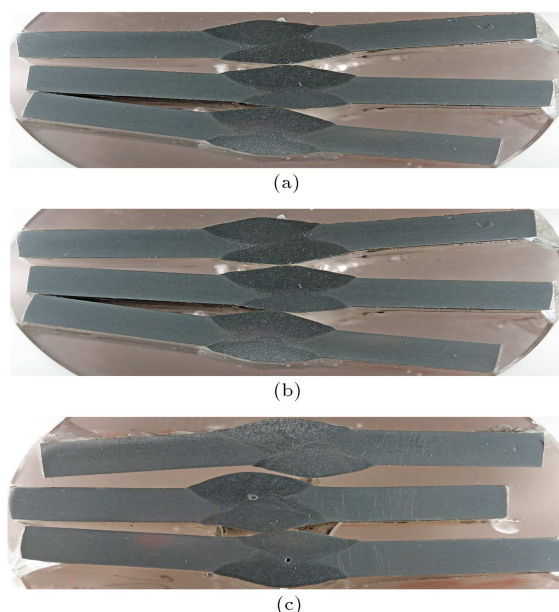


Figure 3. Macrostructures of (a) untreated (UT), (b) 12 h CT, and (c) 24 h CT.

shielding, unstable arc, short arc gap, and poor welding technique [24]. Although porosities are seen only in two samples, the rest are perfectly joined and defect-free. Thus, it can be claimed that correct parameters and good welding techniques have been adopted during the welding process.

Thus, Figures 4–6 show optical micro-graphs of welded AA5083-base metal with AA5356 filler metals. In the 5356 weld metals, dispersed and crystallized particles of several micrometers in size are observed. The composition analysis of the crystallized particles using EDS revealed that those of the base metal were mainly composed of Al, Fe, and Mn, whereas those of the weld metals were mainly Al, Si, and Mg. However, weld defects, such as blowholes and deposited metal cracking, were not detected by means of screening test

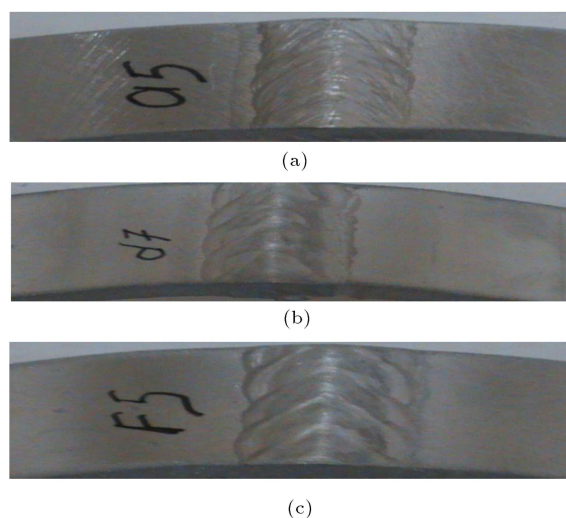


Figure 2. Samples after bending test: (a) Untreated (UT), (b) 12 h CT, and (c) 24 h CT.

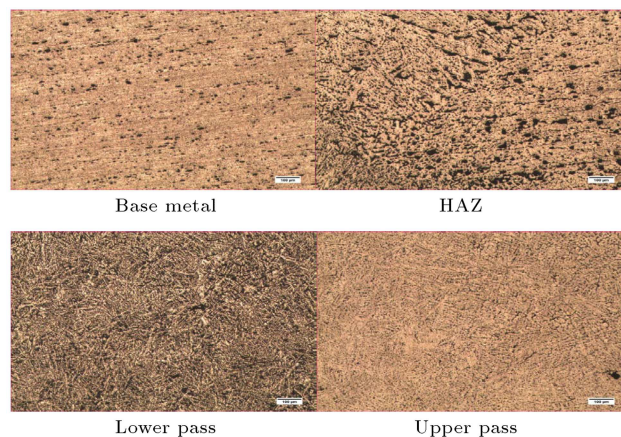


Figure 4. Micro-graphs of welded UT sample with different zones.

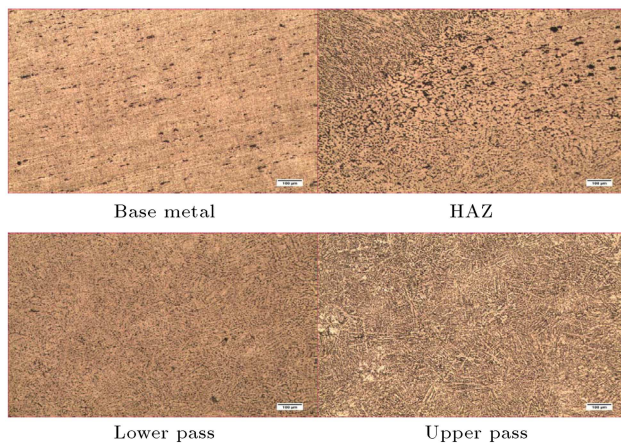


Figure 5. Micro-graph of welded 12 h CT sample with different zones.

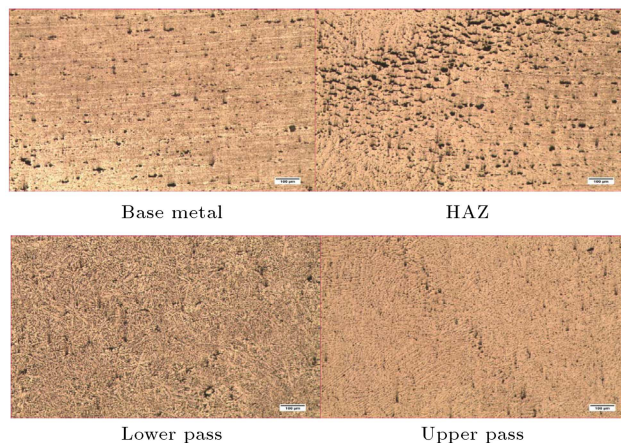


Figure 6. Micro-graph of welded 24 h CT sample with different zones.

or microscopy in the weld metals. It is seen that better distribution of precipitates and particles is evident in these pictures (Figures 4-6). Cryogenic treatment significantly refined the microstructure. Though, these are just preliminary work, further studies need to be conducted to address the reasons for better microstructural and mechanical improvements. Thus, different phase transformation and uniform distribution of precipitation resulted in better mechanical responses of the material (i.e., tool steels) [11,12,25].

3.4. Hardness measurements

The impact of cryogenic treatment on strength was studied via micro-hardness tests. Vicker's hardness measurements were taken as 1 mm intervals starting from both sides in going towards the weld centre. Results are presented in Figure 7. Hardness values on the fusion line are the highest, i.e. 72 HV for UT, 87 HV for 12 h CT, and 99 HV for 24 h CT, respectively. It is apparent that hardness values and tensile properties are strongly affected by the sub-zero treatment (Table 4).

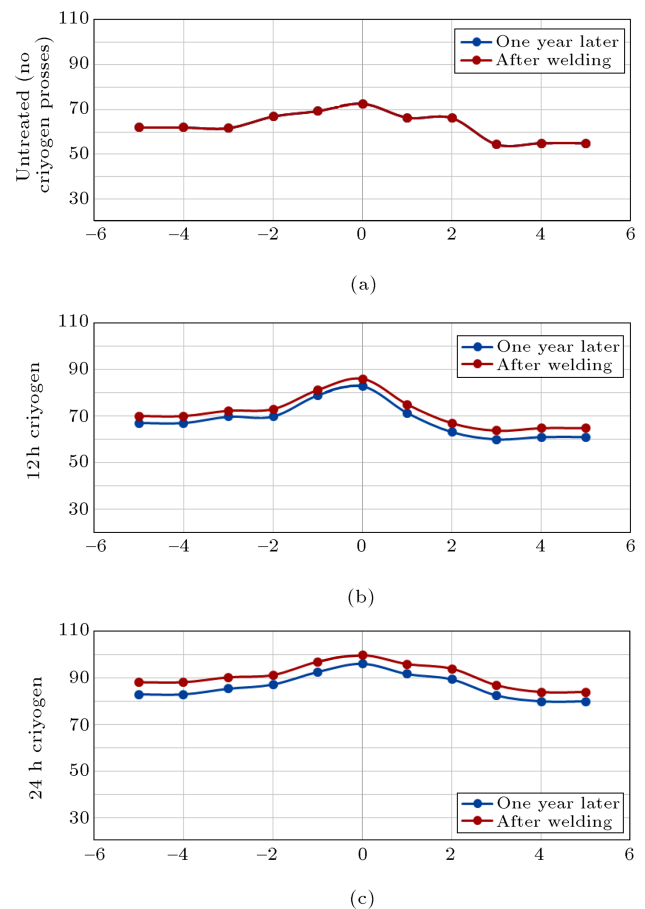


Figure 7. Micro-hardness distribution of welded samples after welding and one year later after welding cryogenics processes: (a) UT, (b) 12 h CT, and (c) 24 h CT.

Similarly, test has been conducted on several samples that were first tested at room temperature for about one year. It is noticeable that hardness values on the fusion line are still the highest, i.e. 72 HV for UT, 82,8 HV for 12 h CT, and 96,1 HV for 24 h CT. A similar trend is also apparently valid for tensile properties (see Table 4). It is unambiguous that changes in the strength values are more or less negligibly small, and the samples soaked at -140°C for about 24 h have turned into finer and more stable structures than that of samples kept for 12 h.

Although a detailed study has not yet been conducted on the microstructural evaluation, it is believed that detailed changes in the microstructure improve mechanical responses of the present welded samples, where for non-ferrous metals particularly, the effect of sub-zero treatment is still unclear.

3.5. Microstructure

Over the years, the term cryogenics has been used to refer to the temperatures around and below -150°C , where it has been mostly used to reduce the amount of retained austenite, particularly in tool steels [26,27]. Thus, Kalsi et al. [27] reported that most steels retained

austenite together with primary and secondary precipitates in the microstructure. Their size and distribution resulted in better mechanical properties, such as wear and fatigue resistances, hardness, and toughness, and also in physical properties (i.e., chemical stability and thermal and electrical conductivity), because sub-zero treatment for soaking time and temperature plays important roles.

Cryogenic treatment has been recently introduced to Al alloys. Although the main mechanisms are still obscure, substantial improvements to mechanical properties are well noticed [28–30]. As a result, several studies on the effects of cryogenics on the behavior of Al alloys have been recently developed [26,31]. According to the laws of thermodynamics, entropy is zero at absolute zero temperature [32]. Therefore, deep sub-zero treatment is mostly usable relieving internal stresses and strains in the material. When the material is kept at extremely low temperatures for elongated durations, the material tends to reach equilibrium conditions [32–35]. It is mostly claimed that microstructure gets finer during the process [36]. Thus, structure at about the welded joint after deep cryogenic treatment can be refined, because there is a great temperature difference during the cryogenic treatment. Internal forces generated during contraction of the bulk create internal stress and so produce some sort of stress concentration in the material. Thus, high-density crystal defect, e.g., dislocations, occurs in the matrix during sub-zero treatment.

In order to reduce the internal energy, dislocations are arranged in such a way that they are mostly aligned and so entrapped along the sub-boundaries. Thus, micro-structure tends to form many fine sub-grains in the matrix forming crystallites and refinement of the grains. As a result, this refined structure contributes to an increase in the strength of the material [37].

Both the sub-structure and elemental distribution become finer during sub-zero treatment. It has been recently noticed that during cryogenic cooling, alloying elements, such as Mg and Cu, become increasingly dense and fine throughout the matrix [36]. Particularly, ageing process right after cryogenic treatment yields Mg and Cu agglomeration at several zones, initially indicating coarse Mg- and Cu-rich zones that are getting finer and so homogeneously distributed within the matrix.

Hence, it is possible to state that during cryogenic treatment, after contraction of the crystal, some of the deformation energies convert into increased internal energy of the alloy. Being now in a meta-stable condition, solid solubility of the alloy is further reduced with decreasing the cryogenic temperature [36]. Thus, for the present material, it is possible to speculate under what condition partial super saturation of the solute elements occurs, where ultra-fine nodules of

precipitate of β phase (Mg_2Al_3) emerge, particularly along the sub-grain boundary lines. Thus, during the period of recovering the sub-zero temperature back to the room temperature level by somewhat ‘*Premature Ageing*’, the content of ultra-fine β phase (Mg_2Al_3) precipitation possibly takes place in previously-aligned dislocation arrays or networks.

Besides, close to welded joints, tensile strength can be further increased due to the formation of very small α (Al) grains and large specific-surface of grain boundary area. Plus, precipitation of nano-sized dispersed β phase plays further roles in the overall strength due to dispersion strengthening. Thus, in the present work, hardness values are considerably higher than those of the sideways material due to the so-called refinement of the structure within the weld zone. The overall strength can obviously be increased by reducing the sub-zero temperature and prolonging the soaking time [37].

In the present work, it has been found that after sub-zero treatment, the samples resting at room temperature condition for one year do not result in any considerable change in the strength of the material. As mentioned earlier, this fact can simply be attributed to the very fine microstructure tailored during the present cryogenic treatment, and its stability against the effect of temperature rises to the level of room conditions. Thus, room temperature level is effective in changing and softening the material, and even keeping it at this level for a long period of time (one year) does not degrade the advanced mechanical strength of this aluminum material.

4. Conclusions

The effect of sub-zero treatment on microstructural and mechanical behaviors of the AA5XXX aluminum alloy welded by MIG technique was studied. According to the findings, the following conclusions can be given as follows:

1. Compared to the untreated material (UT), tensile strengths of 12 h and 24 h sub-zero temperature treated (CT) specimens increase substantially by 12% and 14%, respectively. Surprisingly, percentage of elongation on average also extends by 2%, where during the tests, almost all specimens failed from the zone of HAZ and the weld metal;
2. After bending tests, macro and micro cracks are not observed, where the samples are defect-free, particularly at the vicinity of the weld line. This in fact indicates how the joints are well integrated;
3. It is believed that sub-zero treatment significantly refines the microstructure. Thus, to relieve the material from thermal contractions, sub-microstructure becomes particularly ultra-fine and

homogenously distributed, and possibly stabilized by some sort of second-phase precipitation during the recovery periods of sub-zero temperature level;

4. Average strength of the material is, therefore, strongly affected by the cryogenic treatment. On the other hand, there is no significant difference in the overall strength of the material right after the sub-zero treatment and that of the specimens rested for one year at room temperature after CT.

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Biographies

Hakan Ates Associate Professor was born in Ankara in 1971. He is an Associate Professor in Gazi University, Department Metallurgical and Materials Engineering. He is an international welding engineer and international welding inspector (comprehensive level). He is also vice manager of the Gazi KABTEM application and research center. He has memberships to KATED (Welding Technology Society) and TPMA (Turkish Powder Metallurgy Association). He has many papers on powder metallurgy and welding processes.

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