Effect of sub-Zero Treatment on Ageing of Aluminium Composite

Ahmet Taşkesen, Sinan Aksöz, and A. Tamer Özdemir

***Abstract*—In this study, B4C reinforced 7075 Al composites were fabricated by cold and hot pressing of Al, Cu, Zn and Mg elemental powders. Two different reinforcement rates (10% and 15%) were used for the composites. In order to examine the structural and mechanical properties, different sub-zero and ageing processes were applied to the composites. Sub-zero and stepped ageing treatments were applied after solutionizing at 470 ^{o}C for different time periods. It was found that hardness of the composite was substantially increased and micro structure was changed. Findings show that, general strength of the material significantly increased particularly after solutionization process.**

***Index Terms*—B4C, aluminium composite, sub-zero treatment, ageing.**

I. Introduction

Recently, there has been a substantial increase in the studies on Aluminum (Al) and its alloys due to especially its lower weight to strength ratio [1]-[3]. Lower density, good formability, ability to reach higher hardness values [4] and to increase its mechanical properties by additional processes also played a fundamental role in improving the usage areas of Al alloys [5]. Inclusion of magnesium in Al alloys results in an improved corrosion resistant as well as weight-strength ratio [6]. Moreover, good wettability properties have a favorable influence on microstructure of the alloy [7]. In addition to various ceramic particulates [8], [9], B_4C is also used in the design of the metal matrix composites (MMC) [6], [10] and a better mechanical strength can be obtained [11]. Because of its unique strength and corrosion resistance, Al7075 alloys, which have been used in aviation and space industry, are both used commercially and become the center of interest for research and development Works. In the current studies on age hardenable Al 7075alloys [12], [13], micro structure of the alloy can be improved, and advanced mechanical properties can also be obtained [14]. On the other hand, subzero process techniques have been partially used and studied in order to improve the overall material structure and properties [15]-[17]. Studies on the sub-zero treatments of Al and its alloys have been gradually increased [14], [18].

The aim of this study was to search the capability to increase the strength of Al7075 composite as far as possible. For this reason, composites having two different rates of B_4C (10%, 15%) particle reinforcements were fabricated by liquid phase sintering. Then, different sub-zero and ageing processes were applied. The change in the structural and mechanical properties was examined.

II. Method

A. Ball Milling and Pressing

In order to achieve B_4C reinforced Al7075 based composite structure, B_4C powders having 20 µm average size, Al powders having 20 µm average size, Zn powders having 10 µm average size and Cu powders having 15 µm average size were mixed for 2 hours by using a mechanical grinder. Then, the mixture was cold pressed into the mold under 25 MPa. Later, in order to fill the voids between metal and ceramic powders with zinc, the furnace temperature was kept 540 ^{o}C and composite specimens were fabricated for a half hour. Finally, the mold was kept inside the furnace until 250 ^{o}C furnace temperature and then placed in the open air to complete the cooling. This method was similar to the fabrication route used by previous researchers [6], [19]-[22].

Different reinforcement rates of B_4C inclusions were performed for the purpose of determining the influence of B_4C addition on the mechanical properties of Al7075+B_4C composite (Table I).

Fig. 1. Photograph of test apparatus for composite fabrication.

TABLE I: Chemical Compositions of Fabricated Composites, wt.%

	Al	B_4C	Zn	Cu	Mg
10% MMC	79	10	5	3,5	2,5
15% MMC	74	15	5	3,5	2,5

B. Metallographic Investigations

Test specimens were cut into slices and polished with

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sandpaper in order to make suitable for metallographic and micro hardness examinations. General characteristic of the structure was obtained with the analysis of Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Spectroscopy (EDS).

C. Sub-Zero and Ageing Treatments

After applying solutioning treatments at different time periods, the specimens were subjected to sub zero cooling process at –140^{o}C, and then stepped ageing treatment was applied (Fig. 2a) and Fig. 2b)). Furthermore, conventional T6 ageing treatment was performed for 25 hours prior to sub zero treatment in order to get the effect of sub zero treatment on the hardness of the specimens (Fig. 2c). In Fig. 2a), the specimens, which were firstly subjected to solutioning process at 470 ^{o}C for 12 hours, were treated to sub zero process at 140^{o}C for 24 hours, and then they were aged at 120 ^{o}C for every 3 hours. Quenching process was applied after every 3 hours for the stepped ageing process. In Fig. 2b), hardness values were obtained with the similar aging treatments from the test specimen solutionized at 470 ^{o}C for 2 hours. 3 hour stepped ageing process was applied for the determination of the maximum hardness value. Test specimens were subjected to 9 hours ageing process after the sub zero cooling process since the maximum hardness value was obtained for 9 hour-ageing treatment, as seen from Fig. 2b. In addition, in order to see the influence of sub zero treatment on the ageing process without taking into account the solutioning time, a typical ageing process, which was generally applied to Al7075 alloys, was also applied to the test specimens (Fig. 2c)).

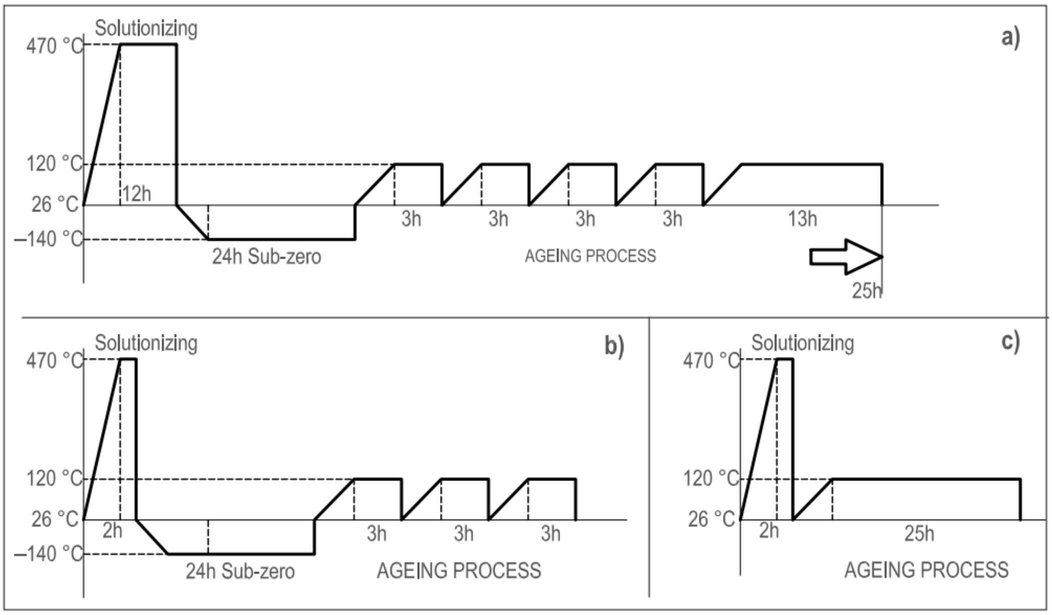


Fig. 2. Diagrams of Applied Sub-Zero and Ageing Treatment a) 470 ℃ +12 hours solutionizing + 24 hours Sub-Zero + ageing at 120 ^{o}C with 3 hours intervals, b) 470 ℃ + 2 hours solutionizing + 24 hours Sub-Zero + ageing at 120 ^{o}C with 3 hours intervals, c) Typical ageing process for 7075 Al alloys.

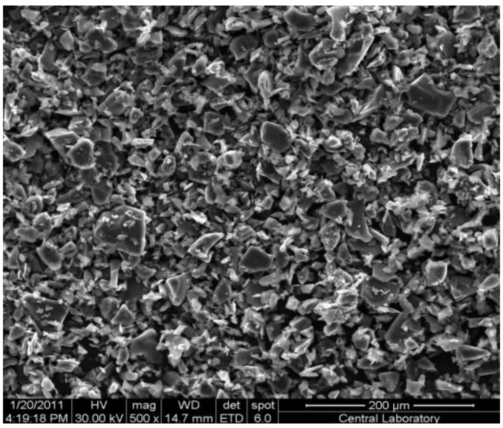


Fig. 3. SEM image of the B4C powders.

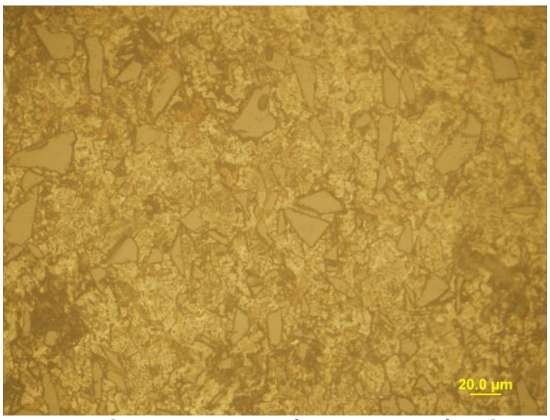


Fig. 4. Optical microscope views of 15 % B4Cp reinforced MMCs produced by liquid phase sintering method

D. Hardness and Density Measurements

After the sub zero cooling and different ageing processes, micro and macro hardness values of the composites were measured. Micro hardness values were used and analyzed since approximately same values were measured for micro and macro hardnesses. Measurements were carried out with SHIMADZU micro hardness device under 10 g and 1 kg loads.

E. Micro Structure of Composite Material and Its Properties

It could be observed from the morphology presented in Fig. 3 that B_4C ceramic powders are angular and agglomerated. Optical microscope images of the hot pressed powders show that a homogeneous distribution and high density can be seen due to the effect of pressing and liquid phase sintering (Fig. 4).

III. Results and Discussion

Existent literature studies stated that subzero heat treatment could be applied especially for steel materials and that this treatment enhanced the strength in particular toughness, corrosion resistance and fatigue properties [15]-[17], [23]. Heat treated material properties can be significantly affected by subzero level as well as the rate of temperature decreasing and holding time of lowest temperature [23]-[26]. Subzero process is theoretically based upon 3rd law of thermodynamics [23]. As it is known, any material kept at 0^{o}K for a long time reaches an ideal equilibrium condition and get rid of structural defects and all internal stress and strains. Furthermore, atomic distances become smaller, particle shape and structure refines, general micro structure gradually becomes uniform and entropy of the material approaches to zero as far as possible. Ultimately, in addition to above mentioned properties, the super arranged microstructure can have high friction and wear resistance as well as high resilience properties [23].

In the first stage of this study, the test specimens, solutionized at 470^{o}C for 12 hours, were subjected to a cooling process at –140^{o}C for 24 hours and then they were gradually aged (Fig. 2a)). In Fig. 5, hardness values of the test specimens, which were solutionized at 470 ^{o}C for 12 hours and subjected to sub zero cooling treatment, can be seen depending on the ageing time. Zero (0) time on the horizontal axis means that the specimens were solutionized at 470^{o}C for 12 hours and then they were subjected to only sub zero cooling treatment at –140^{o}C for 24 hours. Maximum hardness value of the specimens was obtained after approximately 9-hour ageing process. Moreover, hardness seriously increased with increasing B_4C reinforcement rate. SEM images of 10%B4C reinforced composite structure homogenized at the same conditions, with sub zero treatment and without sub zero treatment, are presented in Fig. 6. From the microscopic examinations, it was observed that cracks were formed at the structure (Fig. 6b) and also that micro cracks increased with more than 15%B4C reinforcement rate (Fig. 7).

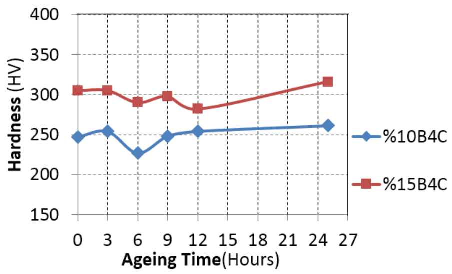
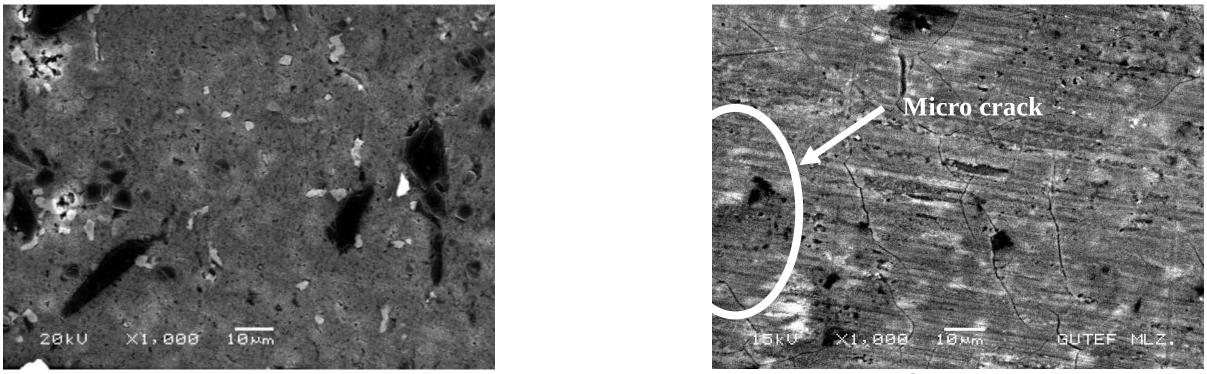


Fig. 5. The effect of 470 ℃ +12 hours solutionizing + 24 hours Sub-Zero ageing treatments on the hardness.

Past studies pointed out that subzero cooling rate might be important and also that micro cracks can be observed with sudden temperature variations [27]. In this work, some cracks were observed as seen in Fig. 6 and Fig. 7. Although these cracks were caused from the subzero treatment, they increased with increasing reinforcement element. Because of this, it would appear inevitable that at least the cracks were formed at some locations of the structure due to very hard nature of the composite material.



a) b)

Fig. 6. SEM micrographs of 10% B4C reinforced structure with and without sub-zero treatment.
a) 12 hours solutionizing at 470 ℃ + water quenching + 25 hours ageing at 120 ^{o}C.
b) 12 hours solutionizing at 470 ℃ + sub-zero treatment + 25 hours ageing at 120 ^{o}C

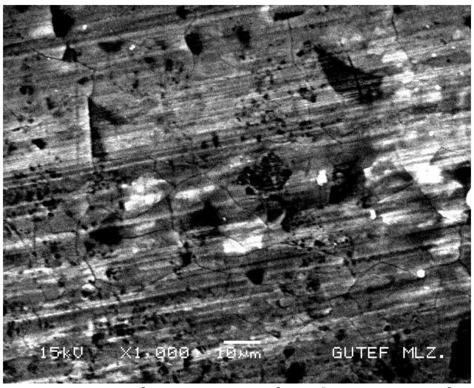
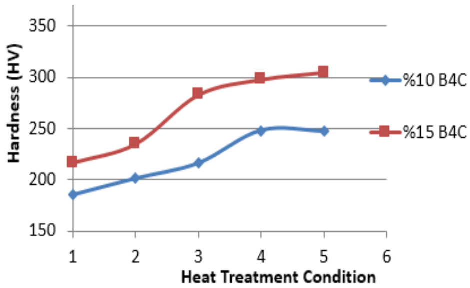


Fig. 7. Micro structures of 15% B4C reinforced composites after 12 hours solutionizing at 470 ℃ + sub-zero treatment + 25 hours ageing at 120 ^{o}C.



1 = Untreated specimen (No heat treatment was applied)
2 = Solutionized at 470 ^{o}C for 2h + 25h aging process
3 = Solutionized at 470 ^{o}C for 2h + subzero cooling + 9h aging process
4 = Solutionized at 470 ^{o}C for 12h + subzero cooling + 9h aging process
5 = Solutionized at 470 ^{o}C for 2h + subzero cooling process

Fig. 8. The effect of heat treatments on hardness.

In order to see the effect of subzero process on the hardness directly, hardness values of untreated specimen and the specimens treated to different applications (for example; solutioned at 470 ^{o}C for 12 hours and 2 hours) were compared. By looking into the hardness values presented in Fig. 8, increasing the solutionizing time caused the alpha (α) phase in the structure become more homogenous and thus the hardness increased more specifically. Moreover, the typical characteristic of the Fig. 8 is that the hardness values obtained from solutionizing + subzero cooling processes is quite higher than the hardness values obtained from classical ageing process.

From the general EDS analysis given in Fig. 9 and Fig. 10, it could be seen that there was an expected B and C density in the structure.

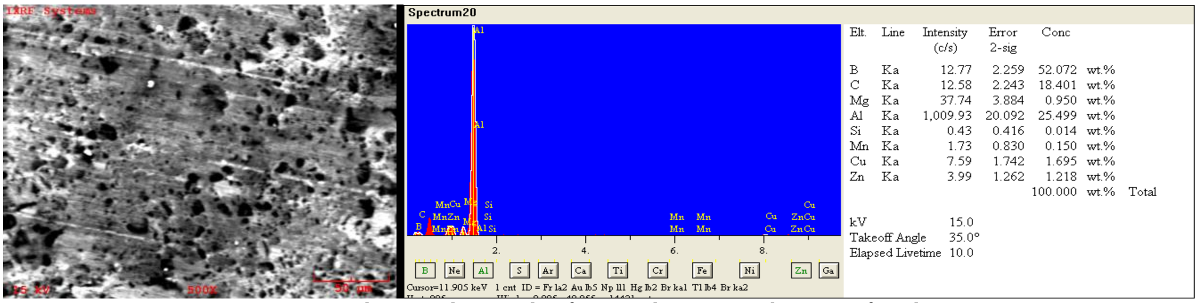


Fig. 9. SEM and EDS analysis results of untreated structure with 10% reinforced B4C.

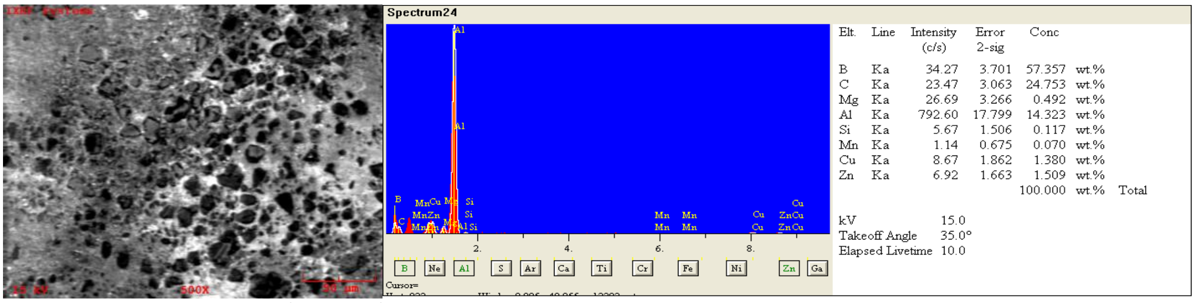


Fig. 10. SEM and EDS analysis results of 10% reinforced B4C composite heat treated as; 12 hours solutionizing at 470 ℃ + sub-zero + 25 hours ageing at 120 ^{o}C.

IV. Conclusions

In this study, the effect of cryogenic treatment on the ageing behavior of B_4C reinforced 7075 aluminium alloy was investigated at the ageing temperature of 120^{o}C. Properties of B4C reinforced Al7075 composite improved by applying subzero treatments at different stages. More homogenous structure was obtained. Moreover, it was found that general strength of the material significantly increased. Findings show that, particularly after solutionization, cryogenic treatment before ageing yielded high hardness values.

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