The effect of cryogenic treatment on ageing behaviour of B$_4$C reinforced 7075 aluminium composites

A. Taşkesen[1]*, S. Aksöz[2], A. T. Özdemir[3]

[1] *Gazi University, Department of Industrial Design Engineering, 06500 Teknikokullar, Ankara, Turkey*
[2] *Gazi University, Department of Machine and Metal Technologies Cubuk, Ankara, Turkey*
[3] *Gazi University, Department of Metallurgy and Material Engineering, 06500 Teknikokullar, Ankara, Turkey*

Abstract

In the current study, an effect of cryogenic treatment on the ageing of a B$_4$C particulate reinforced 7075 aluminium alloy was investigated to identify changes in hardness with respect to ageing time. To achieve this, first, 10, 15, and 25 % by weight B$_4$C particulate containing 7075 Al composite specimens were produced by hot pressing of Al, Cu, Zn, and Mg elemental powders. Next, cryogenic treatment and multiple steps of tempering were adapted to several samples. Such as, after solutionizing at 470 °C and sub-zero treatment at –146 °C, successive intervals of artificial ageing at 120 °C for different time periods were applied. During the heat treatment, hardness values were measured after each interval. Findings reveal that, particularly after solutionization, cryogenic treatment before ageing yields high hardness values. However, the hardness of the composite material exceeds this level during ageing treatment, mainly after long ageing times.

K e y   w o r d s: B$_4$C particles, aluminium composite, sub-zero treatment, cryogenics, ageing

1. Introduction

There is a substantial increase in the studies on aluminium (Al) alloys mainly concerning to improve the lower weight-to-strength ratio [1–3]. Lower density, good formability, ability to reach higher hardness values [4] and to increase mechanical properties by additional processes play an increasingly fundamental role in the usage of Al in aeronautics and defence industry [5]. Particularly, the addition of magnesium (Mg) in Al alloys results in the improved weight-to-strength ratio as well as good corrosion resistance [6]. Also, good wettability properties have a favourable influence on the resulting microstructure of the alloys [7]. On the other hand, among those other traditionally selected ceramic particulates [8, 9], B$_4$C is mostly preferred in the micro-designing of Al-based composites to attain higher mechanical strength levels [6, 10, 11]. Compared to Al 2XXX series, strength and corrosion resistance of Al 7XXX alloys are mostly used commercially in aircraft and space industry and become the main concern of in-

terest of researchers for many years. In the latest current studies, particularly on 7075 alloys [12, 13], the microstructure can further be improved by several techniques, where advanced mechanical properties can be attained [14]. On the other hand, cryogenics is the science of production and effects of very low temperatures. Over the years the term cryogenics has been used to refer to the temperatures approximately around and below –150 °C [14, 15]. Cryogenic treatment has recently been introduced to Al alloys. Although main mechanisms are still ambiguous, considerable improvements on mechanical properties are well noticed [16–18]. It is because of this, several works based on the effect of cryogenic treatment on the behaviour of Al alloys have been gradually developing [14, 19].

In the present work, the aim is to investigate the effect of cryogenic treatment on the ageing behaviour of B$_4$C reinforced 7075 aluminium alloy over a range of 10–25 wt.% of carbides at the ageing temperature of 120 °C. Thus, the main concern is to determine the strengthening capability by introducing a bi-model

---

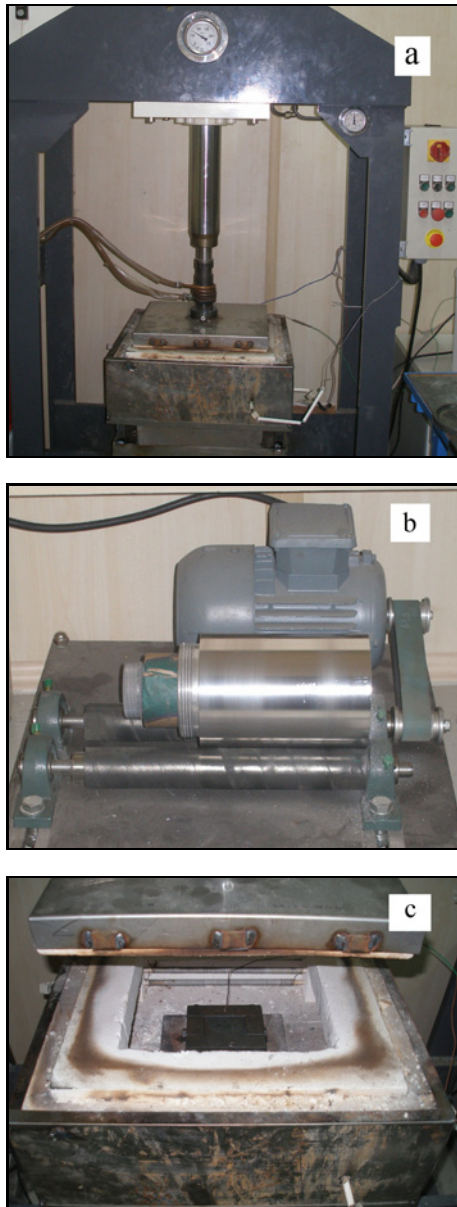*Corresponding author: tel.: +905442939793; fax: +903122120059; e-mail address: taskesen@gazi.edu.tr

Fig. 1. Photographs of test apparatus for composite fabrication: (a) mixing device; (b) press; (c) mould.

distribution of reinforcing carbides and ageing precipitates within the structure of these composites.

2. Experimental procedure

2.1. Manufacturing the material

The necessary samples of the composite material used in this work were manufactured by using the standard techniques of powder metallurgy. The main powders as starting material were supplied from the market. To produce the necessary B_4C particulate reinforced Al7075 composite to be used as the start-

T a b l e 1: Chemical composition 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
25 % MMC	69	25	5	3.5	2.5

ing material in this work, B_4C and Al powders having $20\,\mu m$ average size, Zn powders of $10\,\mu m$ average size, and Cu powders with mean size of $15\,\mu m$ were all mixed for about 2 h by using a mechanical grinder (Fig. 1). Thus, metal and ceramic powders were first weighed with Symmetry EC4000 electronic balance having 0.1 g accuracy and then uniformly mixed in a mixing bowl. To satisfy homogeneous mixing, the metal and ceramic powders together with 36 pieces of zirconia balls were placed into a tank. After mixing, the powders were packed with aluminium foils and then put into a temperature controlled furnace, where the powder mix within the foil was first cold pressed into a mould under 25 MPa, to eliminate the porosity. Later, the furnace temperature was first kept around $540\,°C$ for 30 min to satisfy the conditions for partial melting and liquid sintering. In this way, atomic Zn was forced to fill up the voids between the metal and ceramic powders. After that, the mould was first kept inside the furnace by cooling very slowly down to $250\,°C$ and then left out in the open air. This method is, in fact, the standard procedure to produce Al-based composites [6, 20–23], where consequently, three different batches of composites with different percentages of B_4C content were produced (Table 1).

2.2. Metallography and hardness measurements

Test specimens were cut into slices and polished with sandpaper. In order to make them suitable for metallographic examination and microhardness tests, all specimens were then etched by using a modified Keller's solution. To characterize the microstructures of the products, techniques of Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDS), and FASTMAP were used, respectively. In particular, effects of sintering, cryogenics, and subsequent ageing on outcoming microstructures were determined via FASTMAP and EDS analyses. For hardness testing, at the end of each interval during ageing, hardness values were measured. Each time, the mean values of at least five or six measurements were considered. All microhardness measurements were carried out with SHIMADZU microhardness device under 10 g and 1 kg loads.
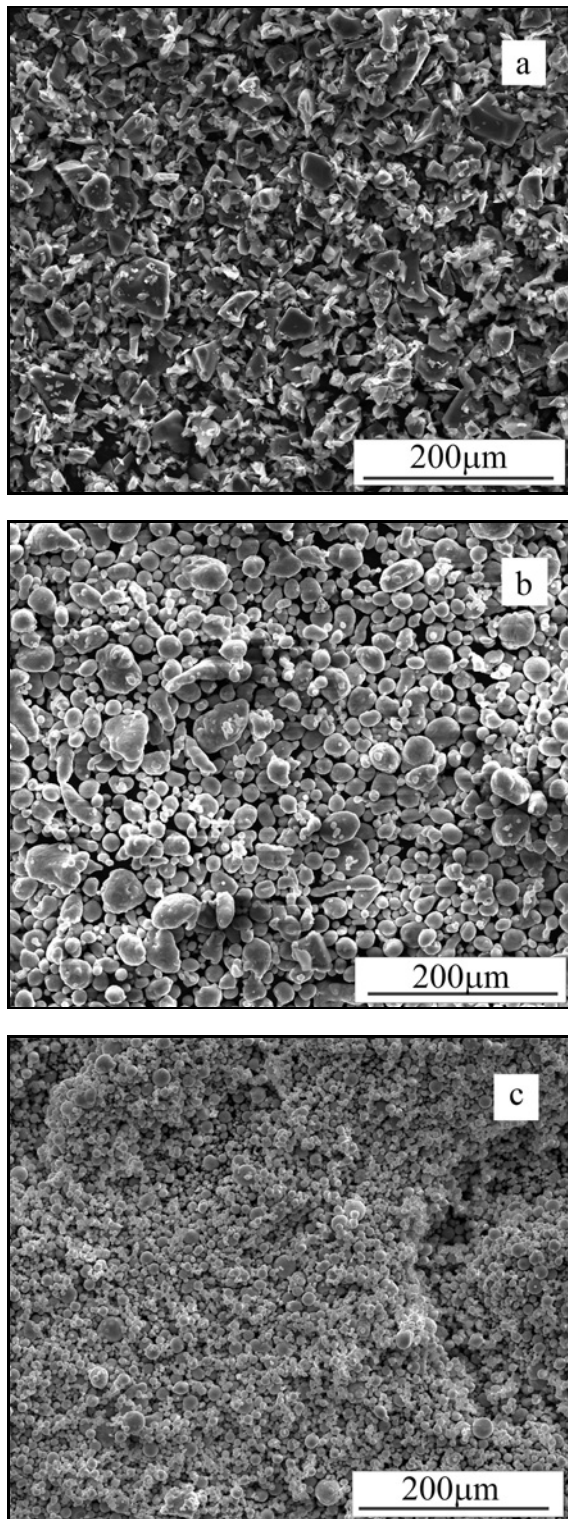
Fig. 2. SEM images of powders: (a) B$_4$C; (b) Al; (c) Zn; (d) Cu.

2.3. Microstructure

As aforementioned, all powders were mixed together with B$_4$C powders at the rates of 10, 15, and 25 % by using ball mill, and then several batches of
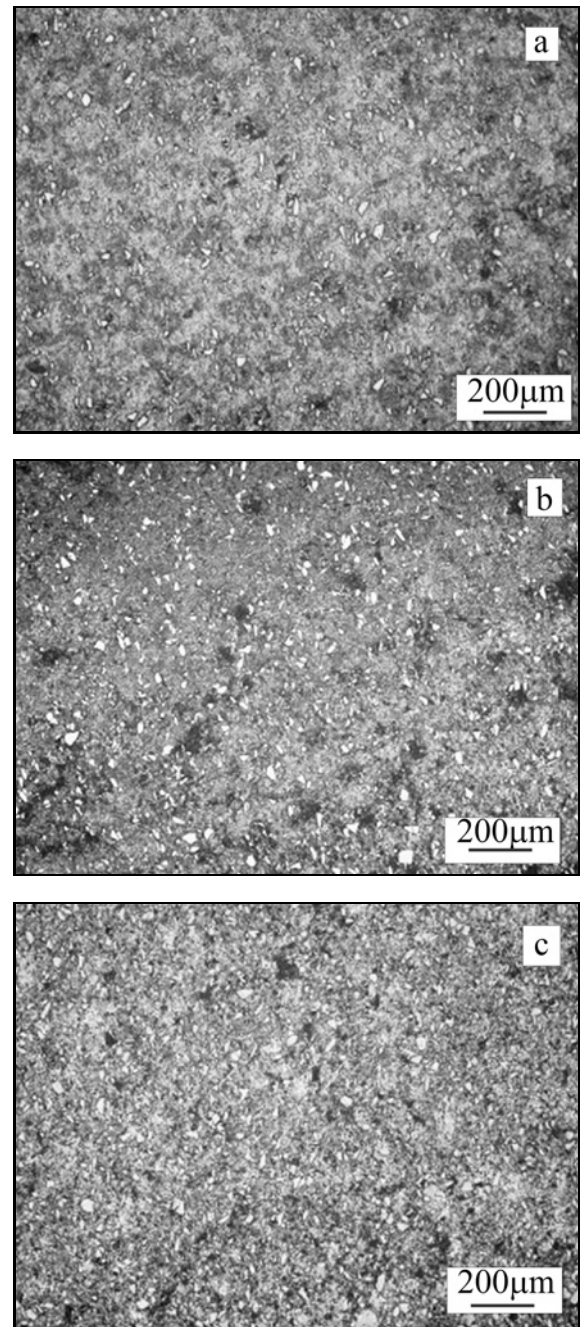

Fig. 3. Optical microscope images of 10, 15, and 25 % B$_4$C reinforced composites after hot-pressing.

composites were fabricated by successive cold and hot pressing processes. The morphologies of the powders are presented in Fig. 2. As apparent from the photographs, aluminium and zinc powders are spherical in shape, while B$_4$C ceramic and copper powders are angular and mostly agglomerated. On the other hand, after hot pressing, a homogeneous microstructure with very high density (low porosity) can be seen due to liquid phase sintering taking place during the process (Fig. 3).
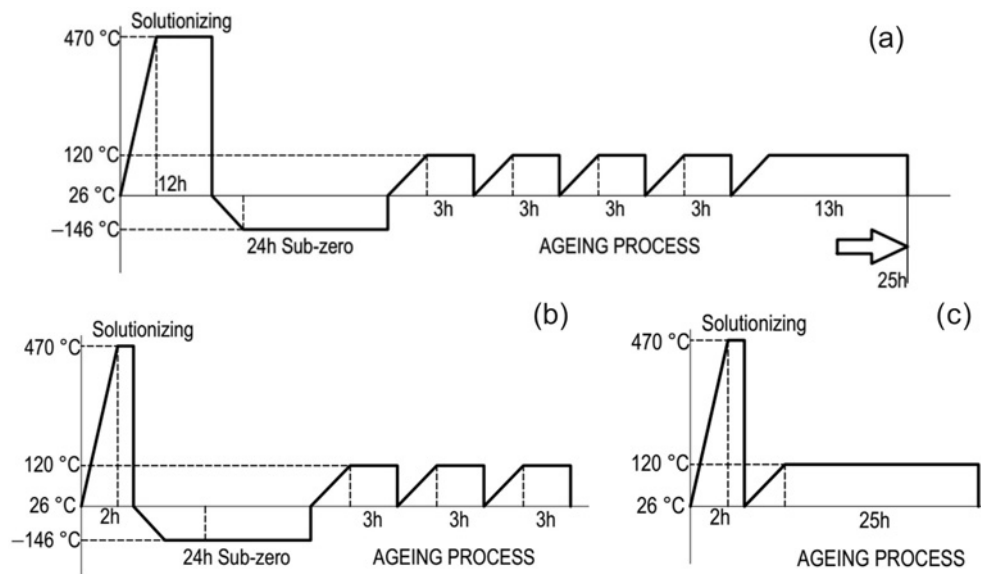
Fig. 4. Diagrams of sub-zero and ageing treatments: (a) 12 h of solutionizing at 470 ℃, 24 h of sub-zero cooling at –146 ℃, and 4 steps of ageing with 3 h intervals, and a final step of 13 h at 120 ℃; (b) 2 h of solutionizing at 470 ℃, 24 h of sub-zero cooling at –146 ℃, and 3 steps of ageing with 3 h intervals at 120 ℃; (c) standard artificial ageing procedure for 7075 Al alloy.

2.4. Heat treatments

After solutionizing for 2 and 12 h, all specimens were initially subjected to cryogenic cooling and then to multi-phase ageing treatments. As illustrated in Fig. 4a, in the first run, specimens were subjected to solutionization at 470 ℃ for 12 h. Thereafter, they were slowly cooled down to –146 ℃ and rested there for 24 h. Finally, they were multi-aged with 4 consecutive steps of 3 h, and finally with a long step of 13 h at 120 ℃. In the second run, after solutionization at 470 ℃ for 2 h, 3 successive steps of 3 h ageing at 120 ℃ were carried out (Fig. 4b). After each step of ageing, specimens were water quenched. These specific ageing processes were in fact organized to increase the available strength of each sample as high as possible. Finally, a standard T6 ageing treatment (25 h) was also performed to create a baseline to see the net effect of cryogenics and other ageing cycles on the material properties (Fig. 4c).

3. Results

In this study, after each heat treatment cycle, hardness measurements were taken to determine the effect of that specific treatment on the mechanical strength of the material. Thus, after each critical cycle of the heat treatment sequence, hardness values were measured and are given in Figs. 5 and 6. In these figures, zero time of ageing stands for the samples that received the basic solutionizing treatment for about 12 h at 470 ℃ and then the cryogenic treatment at –140 ℃
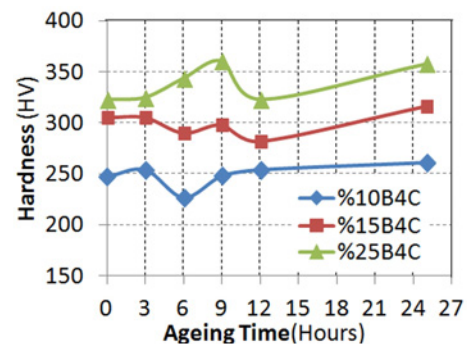

Fig. 5. Variation of hardness after 12 h solutionization at 470 ℃, 24 h cryogenics at –146 ℃, and after each ageing cycle at 120 ℃.

for 24 h. One can see that, as B_4C content increases, the hardness of the material also increases which is an expected result. However, it is clear that during ageing of samples containing 10 % B_4C, after about 3 h, hardness values (250 HV) tend to decrease and at about 6 h reach a minimum of 225 HV. Later, at about 9 h of ageing, hardness increases and achieves the same level of 250 HV. Making a plateau, the hardness almost stays stationary. This trend changes as the percent of B_4C increases gradually. The hardness of the sample having 25 % B_4C considerably elevates to 360 HV, reaching to a maximum peak value at about 9 h of ageing and then quickly drops down to 320 HV at about 12 h of ageing time. After making a zigzag, the hardness then again starts elevating with a constant rate, reaching the maximum value of 350 HV again at about 28 h.
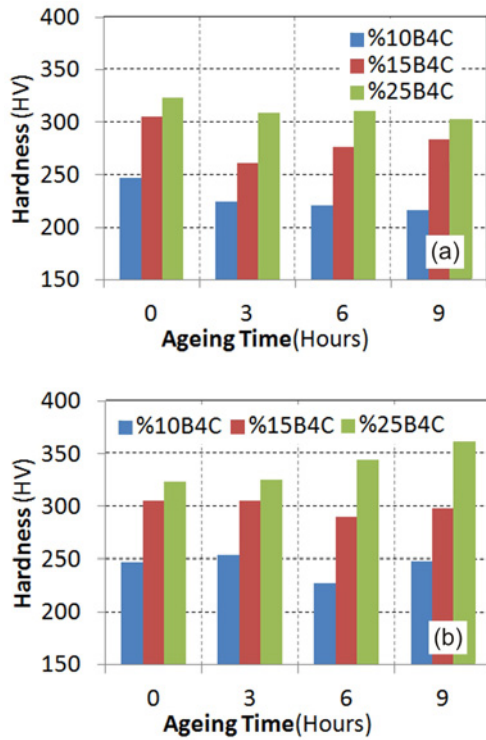
Fig. 6. Variation of hardness after 2 and 12 h solutionization at 470 °C, 24 h cryogenics at –146 °C and after each ageing cycle at 120 °C: (a) 2 h solutionization at 470 °C; (b) 12 h solutionization at 470 °C.
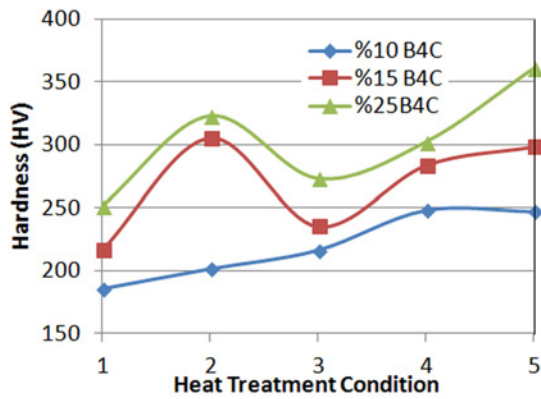

Fig. 7. Effect of heat treatment cycles on hardness. Treatment conditions: 1 – untreated material; 2 – 2 h solutionizing at 470 °C, water quenching, deep sub-zero cooling at –146 °C; 3 – 2 h solutionizing at 470 °C, water quenching, 25 h ageing at 120 °C; 4 – 2 h solutionizing at 470 °C, water quenching, deep sub-zero cooling, 9 h ageing at 120 °C; 5 – 12 h solutionizing at 470 °C, water quenching, deep sub-zero cooling, 9 h ageing at 120 °C.

When all the profiles in Fig. 5 are considered, it is apparent that a two-stage hardening is taking place. Between 3 to 12 h of ageing, a non-steady variation

in hardness exists. Only after 12 h, hardness starts to increase gradually. On the average, in each sample, the first peak hardness value coincides with 9 h of ageing time. Thus, to make a comparison between the results of water quenched, cryogenically treated, cryogenically treated and then aged to the first peak hardness (i.e., 9 h), another graph showing how heat treatment conditions influence the variation in hardness is plotted in Fig. 7. It is apparent that cryogenic treatment before ageing affects the hardness considerably. As a content of B_4C increases, the response of the material to cryogenic treatment also increases. Thus, after cryogenic treatment, the hardness of the samples having 10, 15, and 25 % B_4C by weight, elevates to 240, 310, and 325 HV, respectively. These values are quite high above the hardness values achieved after one-step artificial ageing at 120 °C for about 25 h after solutionizing at 470 °C for 2 h and then water quenching. Instead of water quenching, cryogenic treatment (at –140 °C for 24 h) just before ageing considerably increases the hardness of the samples, right after the ageing process about 9 h, i.e. 220, 280, and 300 HV for 10, 15, and 25 % B_4C reinforced samples, respectively. The time of solutionization has a significant effect on hardness. Instead of 2 h, 12 h of solutionizing at 470 °C makes an increase in the related hardness values. Thus, hardness values elevate to 240, 300, and 360 HV for 10, 15, and 25 % B_4C containing specimens, respectively. This is, in fact, the first peak hardness level reached by the same samples (Fig. 5). After that, hardness values gradually decrease and then more steadily re-elevate to the same high level of hardness. As generally expected, it can be summarized that hardness seriously increases with increasing the content of B_4C reinforcement.

Microscopic examinations, on the other hand, revealed that after cryogenic treatment cracking was readily taking place. Figure 8a shows a crack-free microstructure of the composite having 10 % B_4C particles right after the standard artificial ageing treatment. However, when samples were submitted to cryogenic treatment and then aged under the same conditions, cracking takes place. As presented in Fig. 8b, cracks are visible and confined along some grain boundaries. Figure 9 presents the microstructure of the composite having 25 % B_4C reinforcement. It is clear that as B_4C content increases so does the number of cracks. It is believed that cracks mostly arise during the sub-zero treatment [15]. The increase in the content of reinforcement means the composite becomes increasingly hard and strong. Therefore, after solutionizing and water-quenching, during the process of subsequent deep sub-zero cooling, the material becomes increasingly crack sensitive. This is supposed to be due to thermal contractions during the process of deep sub-zero treatment. Figure 6 implies that solutionization time has an influence on the hardness
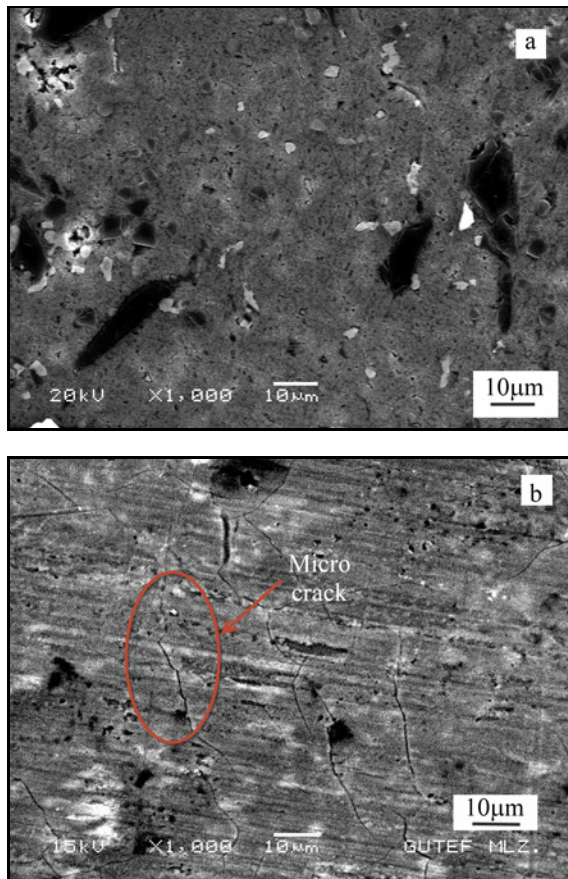
Fig. 8. SEM micrographs of 10 % B$_4$C reinforced structure with and without sub-zero treatment: (a) 12 h solutionizing at 470 °C, water quenching, and 25 h ageing at 120 °C; (b) 12 h solutionizing at 470 °C, sub-zero treatment at –146 °C, and 25 h ageing at 120 °C.
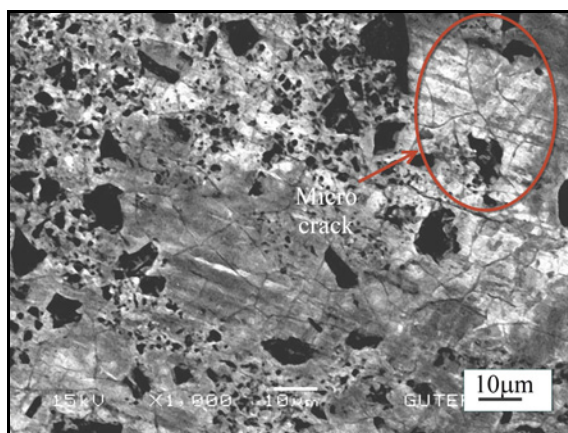

Fig. 9. Microstructure of 25 % B$_4$C reinforced composite after 12 h solutionizing at 470 °C, sub-zero treatment at –146 °C, and finally 25 h of ageing at 120 °C.

of the material. As the solutionization time increases, hardness also increases.

EDS analyses were performed to get detailed information about the progress in microstructure during heat treatment cycles. Figure 10 presents the untreated structure of the 10 % B$_4$C containing sample right after hot pressing. On the other hand, Fig. 11 displays the microstructure of the same material after cryogenic treatment and ageing up for 25 h at 120 °C. It is apparent that the high intensities of the elemental B and C indicate the high content of the B$_4$C particles within the matrix. According to the MAP analyses for the same composite material having 10 % reinforcement, B$_4$C particles are homogeneously distributed, as normally expected (Figs. 12–14). In addition, elements of Cu and Mn also become increasingly dense and fine throughout the matrix. From the SEM images given in the corresponding figures, dark regions represent B$_4$C and Cu zones in the structure. Particularly, ageing process after cryogenic treatment yields Cu agglomeration at several zones indicating that coarse Cu-rich zones are heterogeneously distributed within the matrix (Fig. 14).

4. Discussion

In this study, the role of ageing and in particular the effect of cryogenic treatment on subsequent ageing properties and progress in hardness of the material is in question.

Cryogenics is the science of production and effects of very low temperatures. From this definition, cryogenics means treating the material below the freezing point of water. Over many years, the term cryogenics has been used to refer to a temperature close to –150 °C and below this level. According to the laws of thermodynamics, there is a limit of the lowest temperature that can be reached [24]. At this level, the third law states that entropy is zero at absolute zero temperature. Therefore, deep sub-zero treatment is mostly used to relieve internal stresses and strains in the material. When the material is kept at extremely low temperatures for a prolonged duration, the material tends to develop equilibrium conditions [24–27]. To attain the minimum entropy level, the defects in the material are almost minimized, with refined and uniform grains. Furthermore, atomic distances become smaller, particle shape and structure refines, so the general microstructure gradually becomes uniform. As the material is brought back to room temperature, the defect level still reflects the equilibrium condition. Hence, with this super arranged microstructure, several mechanical properties such as resistance to wear, corrosion, and fatigue enhance considerably as well as high resilience properties [16–18, 24].

In the present work, after the process of solutionization and water quenching, the effect of cryogenic treatment on the hardness of the material is notewor-
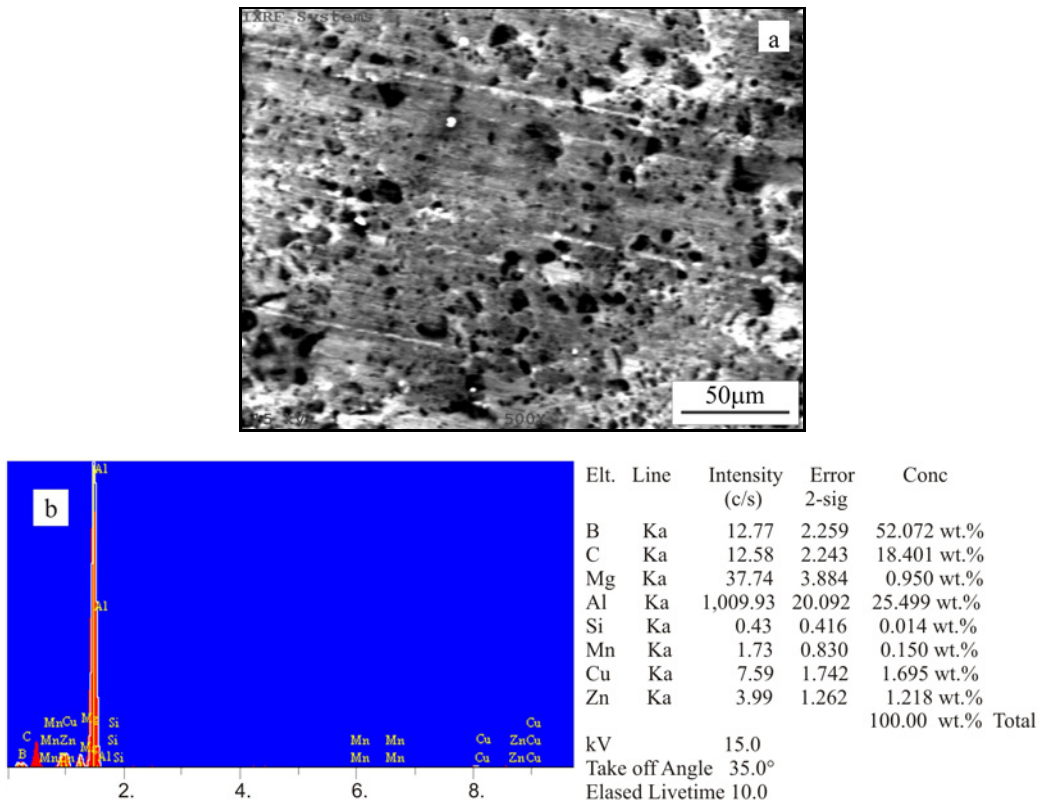
Elt.	Line	Intensity (c/s)	Error 2-sig	Conc
B	Ka	12.77	2.259	52.072 wt.%
C	Ka	12.58	2.243	18.401 wt.%
Mg	Ka	37.74	3.884	0.950 wt.%
Al	Ka	1,009.93	20.092	25.499 wt.%
Si	Ka	0.43	0.416	0.014 wt.%
Mn	Ka	1.73	0.830	0.150 wt.%
Cu	Ka	7.59	1.742	1.695 wt.%
Zn	Ka	3.99	1.262	1.218 wt.%
				100.00 wt.% Total

kV 15.0
Take off Angle 35.0°
Elased Livetime 10.0

Fig. 10. SEM and EDS analyses of untreated sample with 10 % B$_4$C.

Elt.	Line	Intensity (c/s)	Error 2-sig	Conc
B	Ka	34.27	3.701	57.357 wt.%
C	Ka	23.47	3.063	24.753 wt.%
Mg	Ka	26.69	3.266	0.492 wt.%
Al	Ka	792.60	17.799	14.323 wt.%
Si	Ka	5.67	1.506	0.117 wt.%
Mn	Ka	1.14	0.675	0.070 wt.%
Cu	Ka	8.67	1.862	1.380 wt.%
Zn	Ka	6.92	1.663	1.509 wt.%
				100.00 wt.% Total

kV 15.0
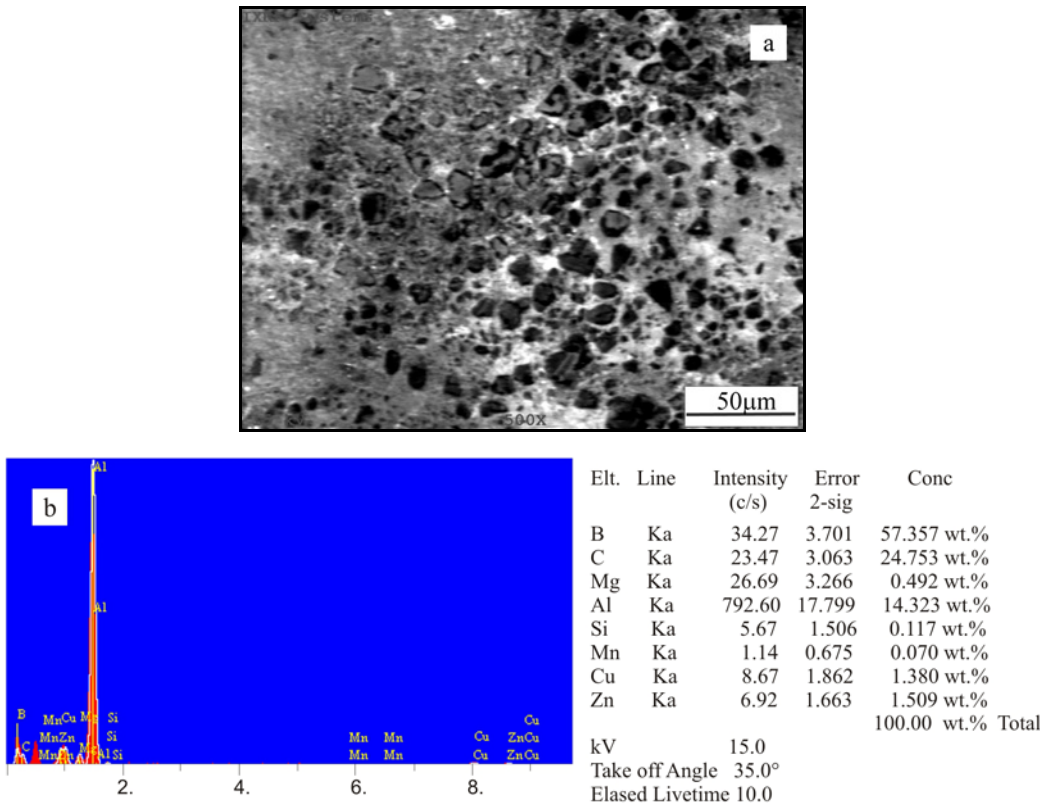Take off Angle 35.0°
Elased Livetime 10.0

Fig. 11. SEM and EDS analyses of 10 % B$_4$C composite after 12 h solutionizing at 470 °C, sub-zero treatment at –146 °C, and 25 h of ageing at 120 °C.
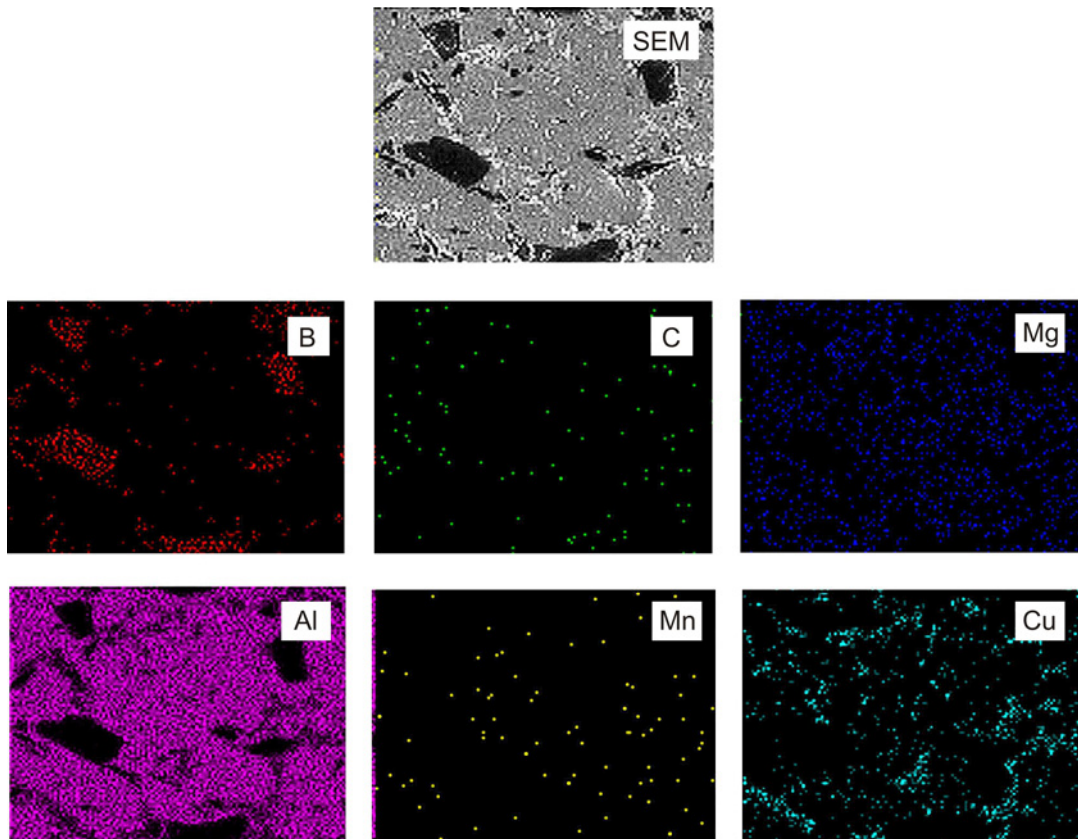
Fig. 12. MAP image of 10 % B$_4$C reinforced composite received standard artificial 25 h of ageing at 120 °C.
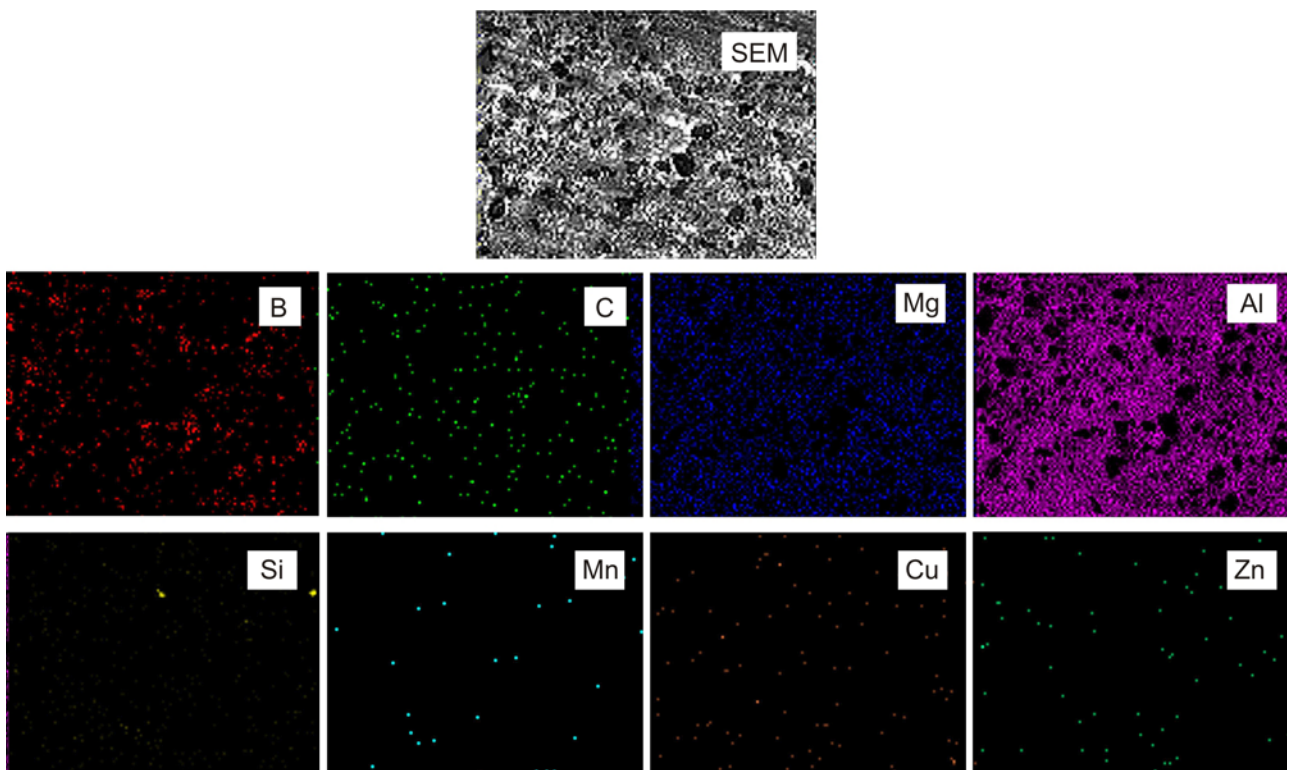

Fig. 13. MAP image of 10 % B$_4$C reinforced composite after 12 h homogenizing at 470 °C and sub-zero treatment at –146 °C.
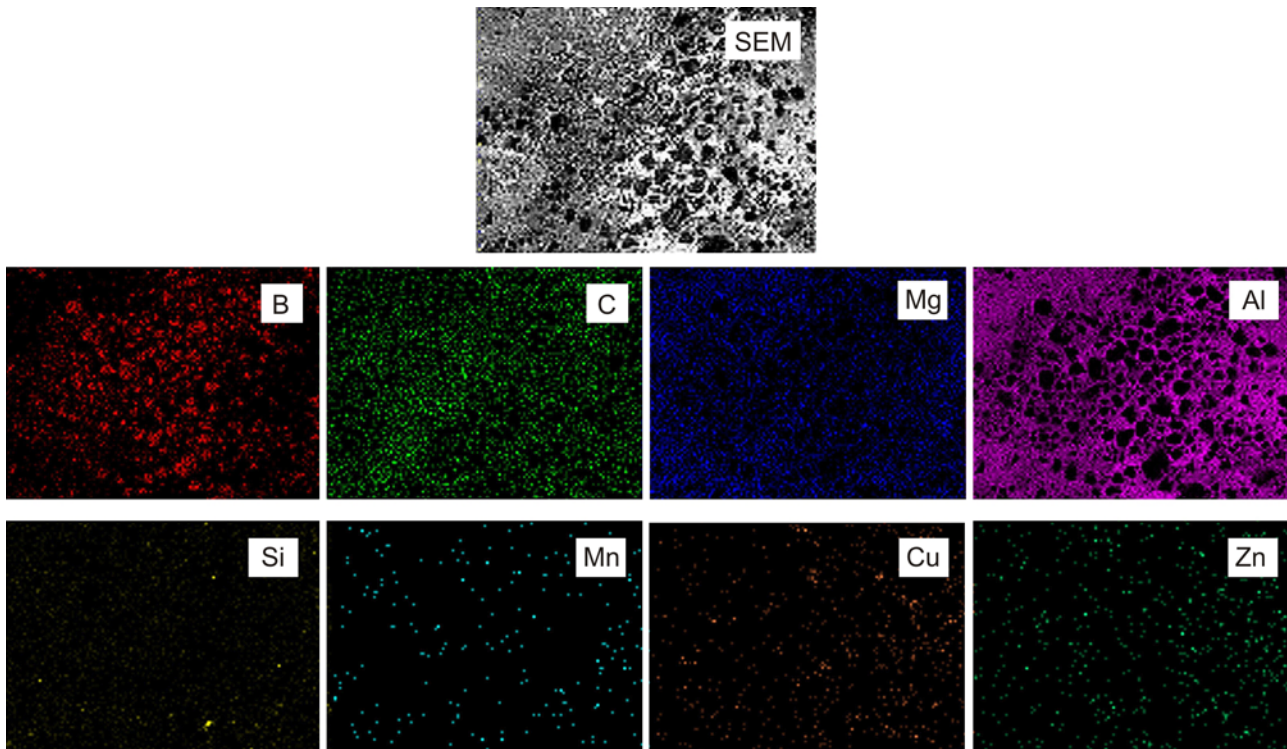
Fig. 14. MAP image of 10 % B_4C reinforced composite after 12 h homogenizing at 470 °C, the sub-zero treatment at −146 °C, and finally 25 h of ageing at 120 °C.

thy (Figs. 5 and 6). Thus, right after the sub-zero cooling, the measured hardness values elevate to 240, 310, and 325 HV for 10, 15, and 25 % B_4C, respectively. To keep this high hardness level or a further increase in the strength of this material, several repetitive ageing stages were adopted right after the process of sub-zero treatment. It is apparent that even right after the application of the sub-zero treatment soon after the solutionizing process, the strength of the composite increases extremely (Figs. 5 and 6). Nevertheless, the sub-zero process was used as an intermediate process instead of a final treatment due to the possibility of the recovery of the material from the state of cryogenics effect and so lose all its strength when at least kept at room temperature. Rather make this increase in strength persistent, after solutionizing and the following sub-zero treatment, ageing process was chosen to be the main consecutive step to increase the hardness at the desired high levels of interest. And rather than using the standard single stage ageing, multiple stages of ageing treatment were preferred to enhance the properties of the material as far as possible.

It is clear from the results that the duration of solutionization treatment has a considerable effect on the outcoming strength of the material after ageing (Fig. 5). Thus, just keeping the sequences of sub-zero cooling and 9 h of ageing at 120 °C the same, but only increasing the solutionizing time from 2 to 12 h, elevates the hardness values from 220 to 250

HV for 10 % B_4C, 280 to 300 HV for 15 % B_4C, and 300 to 360 HV for 25 % B_4C contents. This means the ageing kinetics and formation of coherent precipitates are influenced by the time of homogenization process. Thus, after quenching and cryogenic treatment, the supersaturated single-phase structure becomes monotonous, and elements of Cu, Mn, Mg, and Zn show a more fine and homogeneous distribution at the atomistic scale. Moreover, compared to that of the classical ageing process, quench sensitivity fades away where in return Cu, Mg, and Zn-rich ultra fine and dense precipitates of GP and η' form successively during the next sequences of the ageing treatment. On the other hand, the sudden drop in hardness at the onset of ageing right after the cryogenic treatment is because of the sudden exposure of the material to the annealing temperature (120 °C). Thus, due to temperature effect, the cryogenic microstructure softens rapidly. However, soon after that, ageing dominates and compared to classical ageing treatment, ultra fine particles of GP and η' precipitate homogeneously. The strength of the material increases and finally reaches a maximum after about 9 h of ageing (T6 condition). At this stage, it is clear that both precipitates of (GP ve η') at the nanoscale and ceramic B_4C particles at the microscale, altogether contribute to elevating the strength of the composite. From the point of view of evolution in the microstructure, MAP images given through Figs. 12–14 denote the fine homogeneous el-

emental distribution of Cu, Mg, and Zn. Particularly right after the cryogenic treatment, the distribution of these elements is very fine, dense and homogeneous (Fig. 13). Cryogenic treatment in fact significantly affects the amount of these very fine precipitates and particles, which increases with a long soaking period at cryogenic temperature (24 h at –146 ℃). Thus, it seems that keeping at this low temperature for longer times may directly affect the number of these particles that are the key factors for further improvement in the strength of the present composite material.

On further ageing at 120 ℃ for about 25 h, spots of these elements get large and selective indicating how ageing process proceeds gradually. On the other hand, it is interesting to note that the distribution of elemental B and C within the structure of B_4C particles is also reducing in size during the process of cryogenic treatment. This is an extraordinary result, and there is not any evidence of this observation in the literature. Thus, it might be stated that the structure of B_4C particles was changed during the sub-zero soaking. Actually, it has been reported in some previous studies that several unpredictable structural changes or phase formations and even unknown precipitation sequences can exist in the structure of aluminium, where a novel superfine and homogeneous microstructures in the atomistic level that are not yet fully understood can be encountered [28]. Moreover, there may be several improvements in the structural properties, but no detailed information or explanation about the reason for these changes can be given at the moment [24, 28].

Any property change is mostly attributed to the alterations in the microstructure. According to the past studies, the rate of cooling down to sub-zero level may significantly affect the material properties [15, 29], where some damage in the form of thermal shock cracks may occur if the rate of cooling is not sluggish enough or due to sudden temperature variations [30, 31]. In the present work, the rate of cooling is in the order of 1.5 K min^{-1} and is slow enough to be helpful to avoid any microcracking [15]. It is further stated that investigations would be needed to optimize the impact of this parameter on various types of materials. Hence, it is evident in this work that the crack formation is inevitable, where several cracks are visible after cryogenic treatment (Figs. 8 and 9). They are mostly along the grain boundaries, where they seem to increase in number with increasing the content of B_4C. Because of this, it would appear inevitable that these cracks are mostly formed at the critical sites of the structure due to very hard nature of the composite material.

5. Conclusions

1. Cryogenic treatment uniquely increases the over-

all strength of the 7075 Al/B_4C reinforced composite material. The hardness of the cryogenically treated samples increases considerably compared with that of the composite material right after its manufacturing sequence.

2. Important microstructural changes occur during the initial cryogenic treatment before ageing sequences. Such analyses denote refinement of the elemental distributions of Cu, Mg, and Zn during the cryogenic treatment. Additionally, elemental distributions of B and C within the structure of B_4C particles are also decreasing in size during the process, which is, in fact, an extraordinary result, and there is no explanation for this at the moment.

3. Particularly after solutionizing, water quenching, and then cryogenic soaking, when samples are further aged, the hardness first drops down, but it soon elevates to high values, particularly during prolonged ageing periods at 120 ℃.

4. Multiple ageing treatments enhance the strengthening mechanisms acting during annealing, where bimodal precipitation of the ultra-fine particles rich of Cu, Zn, and Mg seems to be very effective in increasing the overall hardness of the composite samples.

5. As the content of B_4C increases, so does the hardness of the material. Thus, together with the precipitation sequence in the structure, the addition of the B_4C particles to the 7075 matrix seems to work together in the strengthening mechanism of the composite material.

6. Micro-cracking is inevitable. Cracks are mostly along grain boundaries, and they increase in number with increasing the content of B_4C. Even they may occur because of the thermal shocks during sub-zero soaking; they mostly seem to be arising as a matter of the hard nature of the composite material.

References

[1] Bostan, B.: J. Faculty Eng. Architect. Gazi Univ., *23*, 2008, p. 343.
[2] Aksoz, S., Ozdemir, A. T., Bostan, B.: J. Faculty Eng. Architect. Gazi Univ., *27*, 2012, p. 109.
[3] Gokmen, U., Turker, M.: J. Faculty Eng. Architect. Gazi Univ., *27*, 2012, p. 651.
[4] Li, Y., Zhao, Y. H., Ortalan, V., Liu, W., Zhang, Z. H., Vogt, R. G., Browning, N. D., Lavernia, E. J., Schoenung, J. M.: Mater. Sci. Eng. A, *527*, 2009, p. 305. doi:10.1016/j.msea.2009.07.067
[5] Ciftici, I., Turker, M., Seker, U.: Mater.Des., *25*, 2004, p. 251. doi:10.1016/j.matdes.2003.09.019
[6] Taskesen, A., Kutukde, K.: Trans. Nonfer. Met. Soc. China, *23*, 2013, p. 2524. doi:10.1016/S1003-6326(13)62764-8
[7] Contreras, A., Angeles-Chávez, C., Flores, O., Perez, R.: Mater. Charact., *58*, 2007, p. 685. doi:10.1016/j.matchar.2006.11.031

[8] Karantzalis, A. E., Wyatt, S., Kennedy, A. R.: Mater. Sci. Eng. A, *237*, 1997, p. 200. doi:10.1016/S0921-5093(97)00290-6

[9] Wang, H. L., Zhang, R., Hu, X., Wang, C. A., Huan, Y.: J. Mater. Process. Tech., *197*, 2008, p. 43. doi:10.1016/j.jmatprotec.2007.06.002

[10] Ipek, R.: J. Mater. Process. Tech., *162*, 2005, p. 71. doi:10.1016/j.jmatprotec.2005.02.207

[11] Khakbiz, M., Akhlaghi, F.: J. Alloys Compounds, *479*, 2009, p. 334. doi:10.1016/j.jallcom.2008.12.076

[12] Li, J. F., Peng, Z. W., Li, C. X., Jia, Z. Q., Chen, W. J., Zheng, Z. Q.: Trans.Nonfer. Met. Soc. China, *18*, 2008, p. 755. doi:10.1016/S1003-6326(08)60130-2

[13] Harrison, T. J., Crawford, B. R., Janardhana, M., Clark, G.: Procedia Engineering, *10*, 2011, p. 3117. doi:10.1016/j.proeng.2011.04.516

[14] Panigrahi, S. K., Jayaganthan, R.: Mater. Des., *32*, 2011, p. 3150. doi:10.1016/j.matdes.2011.02.051

[15] Kalsi, N. S., Sehgal, R., Sharma, V. S.: Mater. Manufact. Process., *25*, 2010, p. 1077. doi:10.1080/10426911003720862

[16] Reed, R. P., Golda, M.: Cryogenics, *37*, 1997, p. 233. doi:10.1016/S0011-2275(97)00004-0

[17] Bensely, A., Prabhakaran, A., Lal, D. M., Nagarajan, G.: Cryogenics, *45*, 2005, p. 747. doi:10.1016/j.cryogenics.2005.10.004

[18] Darwin, J. D., Lal, D. M., Nagarajan, G.: J. Mater. Process. Tech., *195*, 2008, p. 241. doi:10.1016/j.jmatprotec.2007.05.005

[19] Woodcraft, A. L.: Cryogenics, *45*, 2005, p. 421. doi:10.1016/j.cryogenics.2005.02.003

[20] Acir, A., Turgut, Y., Übeyli, M., Günay, M., Şeker, U.: Sci. Eng. Comp. Mater., *16*, 2009, p. 187. doi:10.1515/SECM.2009.16.3.187

[21] Yilbas, B. S., Khan, S., Raza, K., Keles, O., Ubeyli, M., Demir, T., Karakas, M. S.: Int. J. Adv. Manufact. Tech., *50*, 2010, p. 185. doi:10.1007/s00170-009-2489-6

[22] Taskesen, A., Kutukde, K.: Materiali in Tehnologije/ Materials and Technology, *47*, 2013, p. 169.

[23] Taşkesen, A., Kütükde, K.: Measurement, *47*, 2014, p. 321. doi:10.1016/j.measurement.2013.08.040

[24] Patil, P. I., Tated, R. G.: International Journal of Computer Applications (ICCIA), *9*, 2012, p. 10.

[25] Collins, D. N., Dormer, J.: Heat Treat. Met., *24*, 1997, p. 71.

[26] Yun, D., Lin, X. P., Xiao, H. S.: Heat Treat. Met., *25*, 1998, p. 55.

[27] Lal, D. M., Renganarayanan, S., Kalanidhi, A.: Cryogenics, *41*, 2001, p. 149. doi:10.1016/S0011-2275(01)00065-0

[28] Panchakshari, H. V., Girish, D. P.: J. Eng. Res. Studies, *3*, 2012, p. 105.

[29] Dobbing, D. B.: Metal Forming, *29*, 1995, p. 1.

[30] Preciado, M., Bravo, P. M., Alegre, J. M.: J. Mater. Process. Tech., *176*, 2006, p. 41. doi:10.1016/j.jmatprotec.2006.01.011

[31] Bensely, A., Venkatesh, S., Mohan Lal, D., Nagarajan, G., Rajadurai, A., Junik, K.: Mater. Sci. Eng. A, *479*, 2008, p. 229. doi:10.1016/j.msea.2007.07.035