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ORIGINAL ARTICLE

The Effect of Ultrasonic Cleaning Upon Mechanical Properties of Metal Matrix Composites

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Abstract The aim of this study is to produce composite materials by recycling metallic chips, which are found in industry as a large amount of waste. In addition, it is aimed to investigate the effect of ultrasonic cleaning process as the consolidation behavior and mechanical properties of bulk material directly depend on the cleaning of waste metallic chips. In the present investigation, spheroidal graphite cast iron (GGG-40) was employed as reinforcement material in tin bronze (CuSn10) matrix system. GGG-40 and CuSn10 chips were cleaned by ultrasonic agitation in water for 20 and 40 min. Consolidation of the cleaned metallic chips was achieved with a hot press by applying 820 MPa pressure under 450 °C, and the cylindrical and prismatic metal matrix composite materials with different reinforcement ratios were successfully produced. Energy-dispersive X-ray and scanning electron microscopy analyses were carried out to determine the amount of the oxide removed from the surfaces of chips. The mechanical properties of the samples were determined by hardness, porosity, compression and three-point bending tests. According to the results of the analyses, it was found that CuSn10 surfaces were cleaned from 20%, 50% and GGG-40 surfaces from 35%, 39% oxides during 20- and 40-min cleaning time, respectively. In addition, the results of the mechanical tests revealed that increased ultrasonic cleaning time improves the consolidation quality of metallic chips and it provides successful covering of GGG-40 chips by the CuSn10 chips as a result of a better structural integrity. New machinery parts with high mechanical properties can be produced as a result of recycling of the metallic chips which are available as waste in industry by appropriate cleaning process and this situation makes this study more innovative, economical and environmentally friendly research.

Keywords Cast iron · Bronze · Metallic chip recycling · Ultrasonic cleaning · Hot pressing · Mechanical properties

1 Introduction

Nowadays, it is aimed to reduce the rapid consumption of natural resources and prevent environmental pollution by using recycling methods. In this context, many researches and industrial companies have focused on various recycling techniques for industrial products and machinery components [1, 2]. The investments for this purpose are increasing due to the rise in national and international awareness of the advantages of recycling methods. On the other hand, in order to produce lightweight structures for automotive, aerospace [3, 4] and transpiration industries [5], studies on new porous and composite materials have become widespread [6].

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In industry, large quantities of metallic chips are formed during the material removal process [7]. Copper and iron are frequently preferred materials in industry as a structural material and a large amount of metallic chips are formed as a result of the machining of these materials [8]. In general, these chips are considered to be waste. However, various recycling methods have been developed to utilize these wastes [9]. Gronostajski et al. [9] produced sintered products from aluminum and aluminum alloy chips, which are found as waste in industry. The chips were directly converted into the final products via their production method. They stated that the materials produced by their method exhibit high mechanical properties at room and increased temperatures.

It is reported that the melting and casting process is the most commonly used recycling methods [10], which can be applied to almost any metallic chips. However, these processes require high energy due to the fact that oxidized surfaces of chips result in very low heat and electrical conductivity [11]. Common melting techniques such as induction ovens and electrical resistance ovens have their own low efficiency [12]. Besides, detrimental gases are released during the melting process of chips, and this situation causes environmental pollution.

Recycling of metallic chips like aluminum chips [9], steel chips [10], bronze chips [12], and cast-iron chips [13] and mixed chips such as cast iron and bronze chips [14] was investigated by some authors. Most of these studies were focused on sinterization [15–19] and hot [20]/cold [21] extrusion processes. The proposed method is an alternative to sintering production method since it will not be reached at high temperatures and will be performed at relatively low pressures.

Aslan et al. [22] fabricated metal matrix composite material (MMC) consisting of spheroidal graphite cast iron (SGCI) and bronze (CuSn10) by hot press and investigated their mechanical properties. They successfully recycled metallic chips into machine parts through the hot press unlike conventional melting methods [12]. Salur examined the machinability properties of the metal matrix composites (MMC) consisting of bronze (CuSn10) and cast iron (GGG-40) chips by recycling with double action hot press method. Machinability tests were conducted without using cooling liquid by CNC milling machine and universal turning lathe. The effect of two different feed rates and production parameters on the cutting forces and the surface roughness was investigated [14, 23].

The effects of the chip size on the mechanical properties [24] and microstructure of recycled materials were analyzed by some authors. Most of the works were focused on bronze [10], brass and aluminum chips [11, 25] and aluminum/steel chips [26].

Waste metal chips are usually covered with an oxide layer formed during machining process. Metal cutting fluids also form a soil layer which has adverse effect on the heat and electrical conductivity [15]. It was reported that [13] comparatively small-sized metallic chips [12] and their oxidized surfaces [27] reduced the efficiency of the melting/casting process which results in a material loss by 20% [11].

As stated above, the oxides formed on the surfaces during the formation of chips can lead to poor adhesion and low bond strength in the case of recycling by melting and isostatic hot press. In this respect, it is important to remove the oxide layer from the surface of the chips to recycle and to provide metal–metal contact. In addition, this increases the diffusion bond and the plastic deformation ability of the chips.

Ultrasonic is an effective technique for cleaning various surfaces [28] from delicate removal of [29] particles from semiconductor chips to scaling from steel strip [30] to the removal of oxides.

Major industrial changes in the field of material assembly are observed in the automotive industry, maritime industry and structural links for aviation [31, 32]. These problems can be solved by the control of surface preparation procedures [33]. Ultrasonic and acoustic methods are the most frequently followed approaches since the first stages of structural bonding applications [33].

The most suitable approach to study the bonding strength is to change the surface preparation or surface treatment [34]. Xu et al. [35] utilized ultrasonic wave for removing the precipitates from the surface of TiO_2 nanotube arrays which was covered by debris of titanium oxides. It was also reported that performances of the electrodeposited layer [36], adhesion strength [37, 38], fatigue strength [39], surface morphology [40], tensile stress [41–43] could be enhanced by utilizing the ultrasonic agitation during electrodeposition process.

The literature survey reveals that ultrasonic cleaning can be used especially when cleaning oxides from metallic surfaces. However, no available studies can be found about the cleaning of oxides from the surface of metallic chips.

In this study, the effects of ultrasonic cleaning on the cleanness of the oxides on the metallic chip surface and the mechanical properties of the recycled product were investigated. Metallic chips were first cleaned by ultrasonic agitation in water for two different times, and then these chips were exposed to hot isostatic press to produce cylindrical and prismatic machine parts. The degree of removal of oxide from the chip surfaces was evaluated by EDX (Energy Dispersive X-Ray) analysis and SEM (Scanning Electron Microscopy) analysis. The mechanical properties of produced machine parts were determined by Brinell hardness and compressive strength tests.

2 Materials and Methods

As it is an experimental study, metallic chips used in production are obtained in a controlled manner and no coolant is used during machining. In this way, the chips do not have a quenching effect and chip surfaces are free from coolant residue so that the ultrasonic cleaning efficiency can be monitored more easily.

The surface of metallic chips is covered with an oxide layer during the formation of metallic chips due to high concentration oxygen which occur in high-temperature environment. This situation prevent and/or make difficult the consolidation of metallic chips. Therefore, oxide layers formed on the surface of metallic chips should be cleaned.

In this study, bronze (CuSn10) and spheroidal graphite cast iron (GGG-40) were used for the production of metal matrix composite materials. Production parameters were selected according to previous studies [22, 44] of our research group. Therefore, the production pressure and the temperature were chosen as 820 MPa and 450 °C, respectively (the maximum pressure and temperature). The MMCs were produced by hot press with different reinforcement ratio of cast iron (GGG-40) such as 10 wt.%, 40 wt.%, and these composites were named as 90B10C and 60B40C, respectively. The metallic chips were mixed at a double cone mixer as proposed by [45] and hot pressed as seen in Fig. 1. Total production time reached was 25 min, 10 min for heating at zero pressure and 15 min for waiting under pressure at constant temperature. The same process was repeated for ultrasonically cleaned metallic chips.

As a result of the preliminary experiments, it was determined that sufficient cleaning did not occur in < 20 min, and no significant improvement was observed over 40 min. Therefore, the cleaning time was selected as 20 and 40 min in deionized water and acetone separately. In addition, it was seen in the literature review that these time intervals were appropriate during the cleaning of oxides on the surface with ultrasonic bath [46, 47]. In order to prevent re-oxidation, chips were dried in electrical oven at 120 °C. Ultrasonic cleaner and contaminated water after the cleaning process are shown in Fig. 2.

SEM/EDX analyses were applied to both uncleaned and cleaned samples in order to observe the effect of the proposed cleaning process. Porosity test, hardness test, compression test and a three-point bending test were also carried out to determine the variation in mechanical behavior of MMCs.

The densities of bulk CuSn10 and GGG-40 were known, and the theoretical value of the material should be calculated by using the ratio of the components used in MMCs and the rule of mixture. The number of pores in specimens was determined using the theoretical density values and the experimentally measured density values which were measured with precision scales according to Archimedes balance. The specimen was first weighed in air (X), and then weighed in pure water (Y). The density (q) of the specimen was calculated as follows:

$$q = \frac{X}{(X - Y)} * (q_{\mathrm{w}} - q_0) + q_0 \qquad (1)$$

where q_0 is the density of air and q_{w} is the density of pure water.

3 Results and Discussion

In this section, EDX/SEM analyses, porosity measurements, Brinell hardness values, compression strength and three-point bending tests results were presented. As mentioned in Sect. 2, specimens were named as 90B10C and 60B40C, depending on mixture ratio where B and C letters represent CuSn10 and GGG-40 contents, respectively.

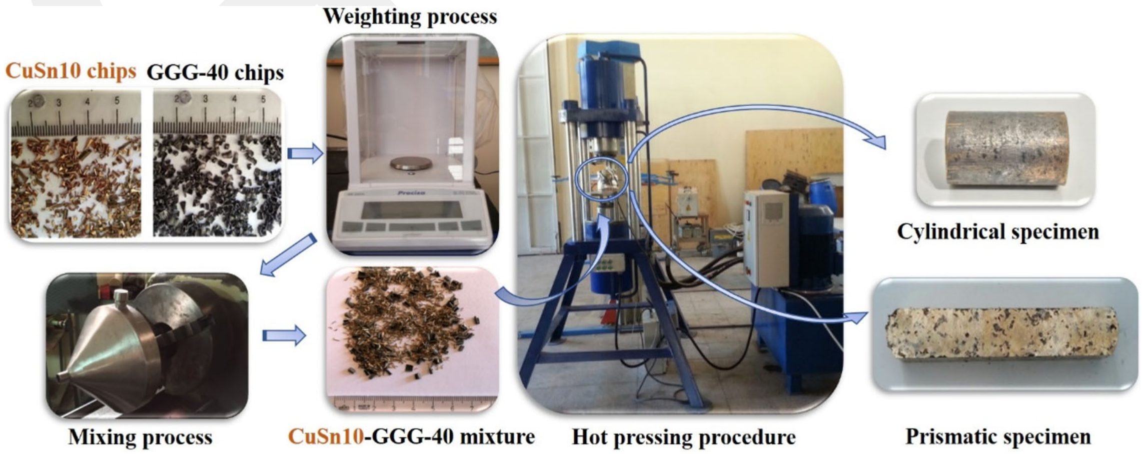


Fig. 1 Schematic view of the production stage

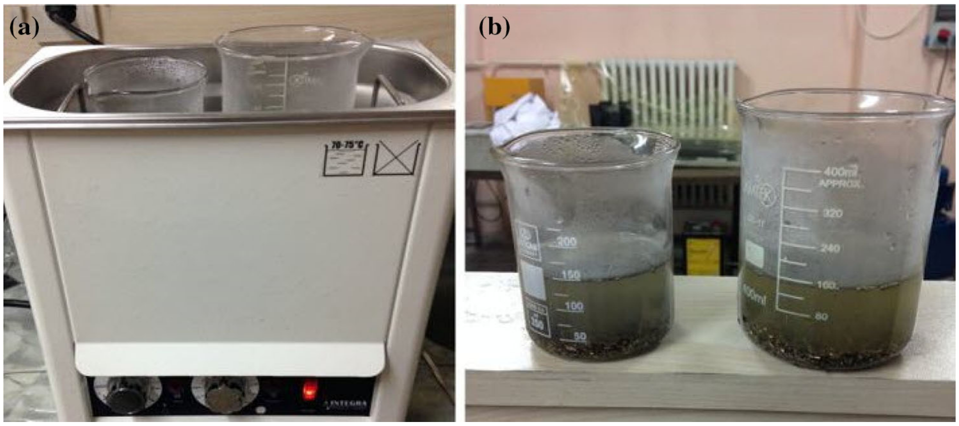


Fig. 2 **a** Ultrasonic cleaning and **b** after the 40 min of cleaning

3.1 EDX and SEM Analyses

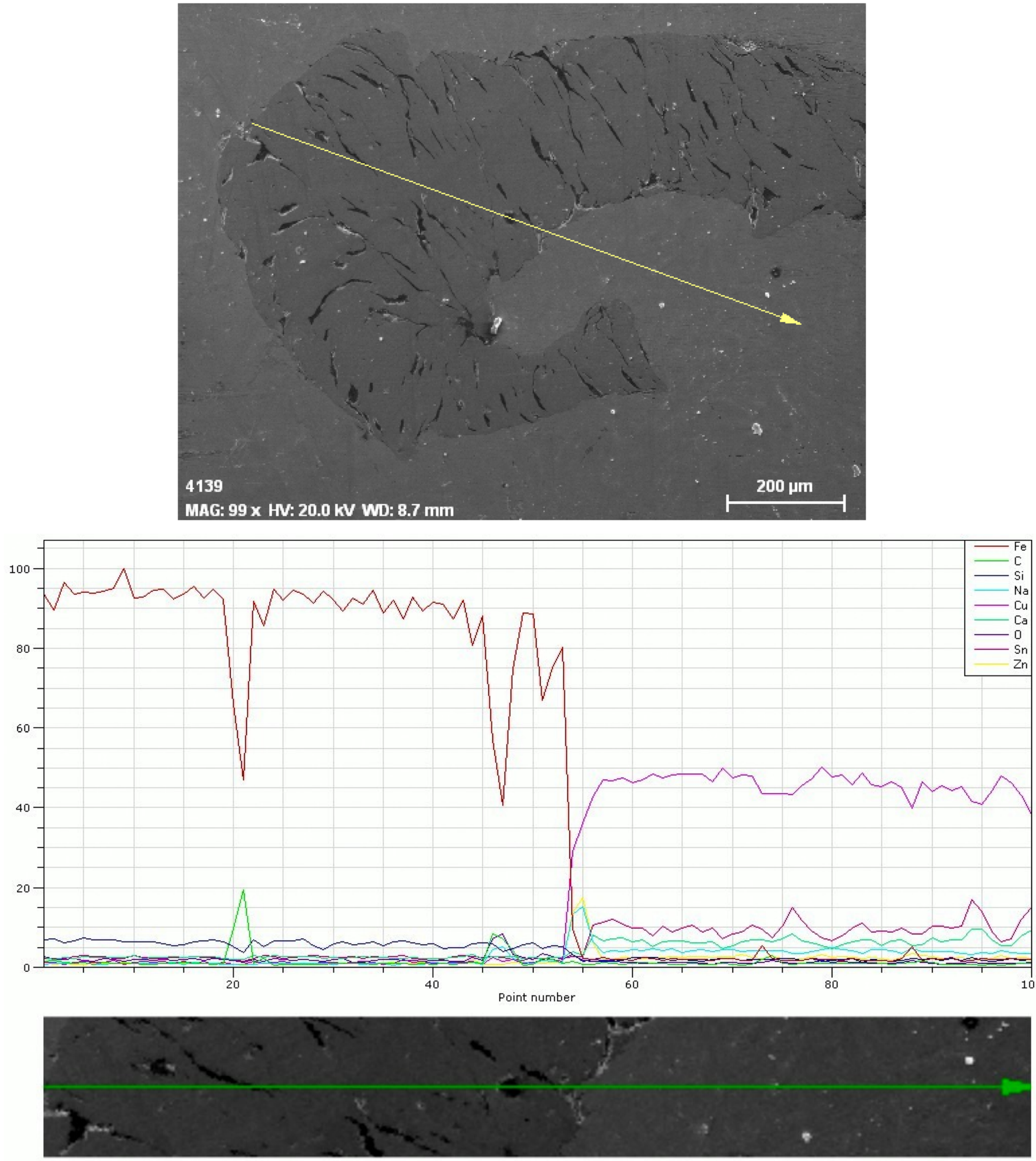
The results of EDX analysis after 40 min of ultrasonic cleaning with cylindrical- and prismatic-shaped specimens are presented in Fig. 3a, b, respectively. The EDX results are taken along the yellow line visible in the SEM image and therefore it does not represent the entire region. The EDX analysis is based on the atomic composition of the material surface. It has been found that oxygen can only be in the form of a compound because the amount of oxygen in this region is reduced and the oxygen does not dissolve in Cu and Fe. In this respect, the oxygen content is considered to be direct oxide content. The chemical concentration is measured from chip boundary region which includes CuSn10 and GGG-40 metallic chips (Fig. 3). Table 1 shows the oxygen content as a function of cleaning time for both CuSn10 and GGG-40 metallic chips.

It is observed that 35–39% of oxides of GGG-40 and 20–50% of CuSn10 metallic chips get removed at 20–40 min of cleaning time as tabulated in Table 1. According to these results, it can be deduced that CuSn10 metallic chips get cleaned more effectively than GGG-40 metallic chips.

Literature review show that several metal oxides located on the metal surfaces can be detected via SEM analysis. It is reported that there can be several metal oxides ranging in size from 150 nm to 1 μm. Also, the geometries of the oxides can vary depending on the formation temperature and how it is formed [48, 49]. Figures 4 and 5 show SEM images of cleaned and uncleaned CuSn10/GGG-40 metallic chips. SEM images support the findings obtained by EDX analysis. It is observed that the oxidized surfaces, which are shown in Fig. 4a, b are cleaned during ultrasonic cleaning process as shown in Fig. 4c–f and bright metallic surfaces can be clearly observed as a result of ultrasonic cleaning. When GGG-40 surfaces are compared to CuSn10 surfaces, despite the fact that there are no clear oxide particles owing to the surface condition of GGG-40, bright and metallic regions are shown as narrow zones of Fig. 5d–f. Both EDX and SEM analyses reveal that oxides and machining debris on the surfaces of metallic chips can be successfully removed from surfaces.

3.2 Porosity and Brinell Hardness Results

Microstructures, porosity measurements and Brinell test results are shown in Figs. 6, 7 and 8 respectively. When Fig. 6a, b is examined, it is seen that the consolidation mechanism of metallic chips is achieved by mechanical interlocking. In addition, it is also observed that no obvious differences have been found in microscale between uncleaned 60B40C (Fig. 6a) and cleaned 90B10C (Fig. 6b) specimens except pores. Besides, XRD test results reported in the previous studies [1] reveal that intermetallic compunds are not observed between the metallic chips and the mechanical interlocking mechanism affects the consolidation. The mechanical interlocking mechanism occurs directly under the control of plastic deformation. CuSn10 metallic chips show appropriate plastic deformation due to high production pressure and temperature and it causes mechanical interlocking. However, oxides on the surfaces of chips restrain the ability of plastic deformation and results in the formation of pores between adjacent metallic chips. In other words, oxide compounds exhibit brittle characteristic [50] because they have directional bonds and/or very complex dislocation structure. Mechanical mismatch emerges when the oxides and easily deformable metal chips are forced to deform together, resulting in porosity during hot pressing [1, 13, 22, 23]. As a result of this situation, GGG-40 metallic chips cannot be completely covered by CuSn10 chips. After the ultrasonic cleaning process, it has been shown that GGG-40 metallic chips are effectively covered by plastically deformed CuSn10 chips. So, it can be concluded that the final machine parts which have been converted from metallic chips have lower pore content. The complete covering of GGG-40 metallic chips

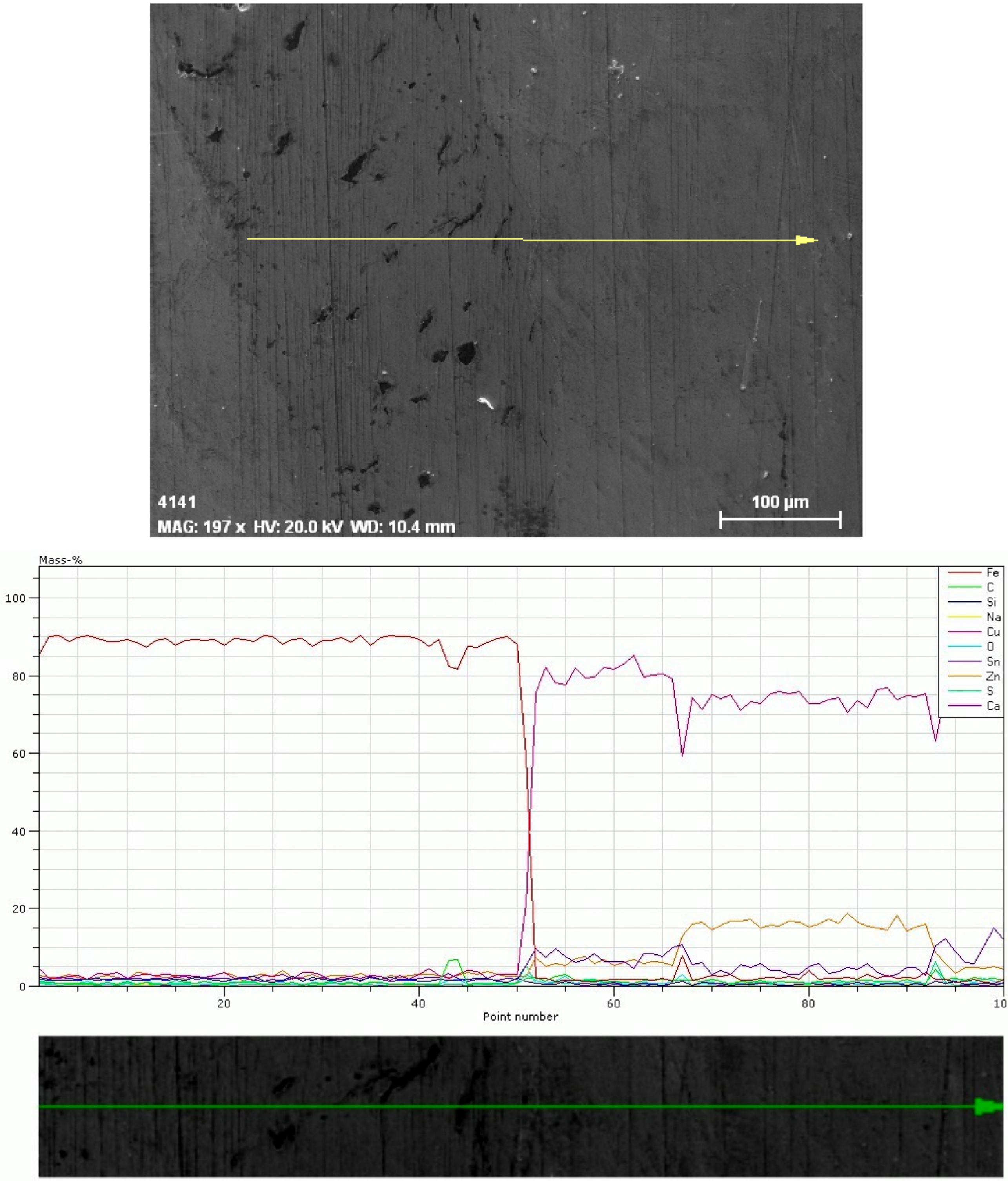


(a) Prismatic shaped specimen

Fig. 3 EDX tests results. **a** Prismatic-shaped specimen. **b** Cylindrical-shaped specimen

causes a decrease in porosity, and quality of mechanical interlocking increases depending on this fact. Strength and quality of structural integrity increase depending on quality of mechanical interlocking, and this fact is supported by some researchers [51, 52].

The porosity test was carried out in accordance with the TS 2350 EN ISO 2738 standard to measure porosity. However, the measurement failed due to the closure of the open pores by the effect of friction between part and die and plastic deformation during hot pressing. Porosity



(b) Cylindrical shaped specimen

Fig. 3 continued

values of cylindrical- and prismatic-shaped specimen of both 90B10C and 60B40C are shown in Fig. 7a, b, respectively. While porosity values of 90B10C decrease at the ratio of 10% as a result of 20 min of cleaning, 40 min of cleaning time results in a decrease in porosity by 53%. It is clearly seen that 40 min of cleaning time is more

Table 1 The effect of ultrasonic cleaning on the amount of oxygen

Ultrasonic cleaning time	at.% Oxygen	
	GGG-40	CuSn10
–	1.98	1.72
20 min	1.91	1.38
40 min	1.21	0.85

efficient than other cases, and this appropriate time provids better mechanical interlocking which consequently decreases porosity in number and size.

Porosity values of cylindrical 60B40C specimen decrease by 4% and 26% based on increase in cleaning time. The decrease in porosity values is related to GGG-40 chips contents. Since the cleaning efficiency of GGG-40 metallic chips is lower than CuSn10 metallic chips, quality of consolidation mechanism decreases, and formation of pore increases.

Prismatic specimen's results are similar to the cylindrical specimen. While porosity values of 90B10C specimen decrease at a ratio of 4% and 23%, 60B40C specimen's values decrease by 3% and 16% based on cleaning time (Fig. 7b). Although porosity values of prismatic specimen decrease as a function of cleaning time, decrement ratio is not as high as cylindrical specimens due to geometrical effects. In other words, the effects of ultrasonic cleaning can be observed more clearly in cylindrical specimens compared to the prismatic specimen. The reason behind this situation can be explained by the fact that the die used for the production of cylindrical specimens allows the metallic chips to show much more plastic deformation due to geometrical effects. As for prismatic die, more pores are formed, and effects of ultrasonic cleaning can be observed restrictedly due to less plastic deformation of CuSn10 metallic chips. Bunlangsup et al. [53] reported that the increased amount of metal oxide may possibly be due to increased oxidation of any remaining alloying elements in the bronze and it may cause an increase in the number of pores.

Brinell hardness test results of cylindrical- and prismatic-shaped specimens are shown in Fig. 8a, b. As for the cylindrical specimen, Brinell hardness values increase by 4–21% for 90B10C and by 4–7% for 60B40C specimen depending on increasing cleaning time. Correspondingly, the hardness values of prismatic specimen increase by 3–14% for 90B10C and by 2–4% for 60B40C. Increment rate of hardness at high GGG-40 content specimen is lower than that of other specimens because of low cleaning

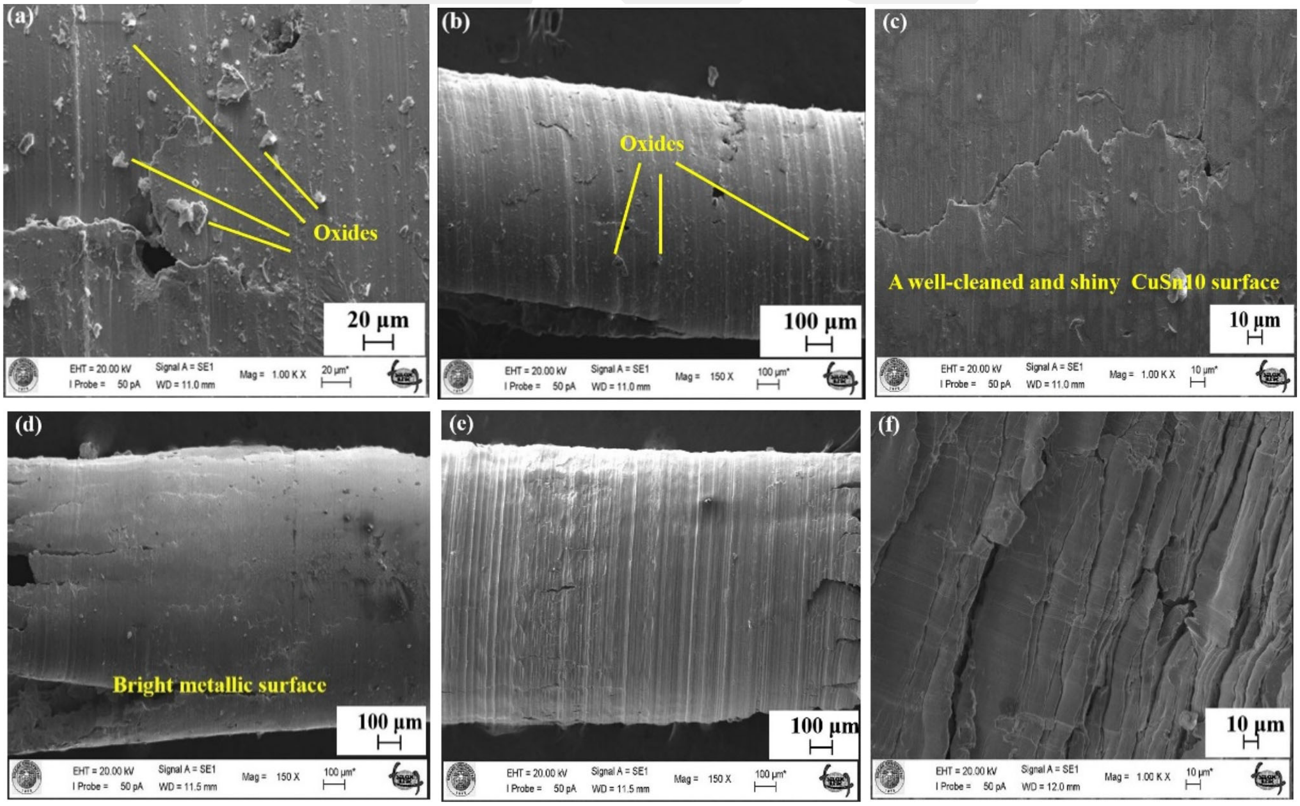


Fig. 4 SEM images of CuSn10 metallic chips **a**, **b** uncleaned; **c**, **d** 20 min and **e**, **f** 40 min

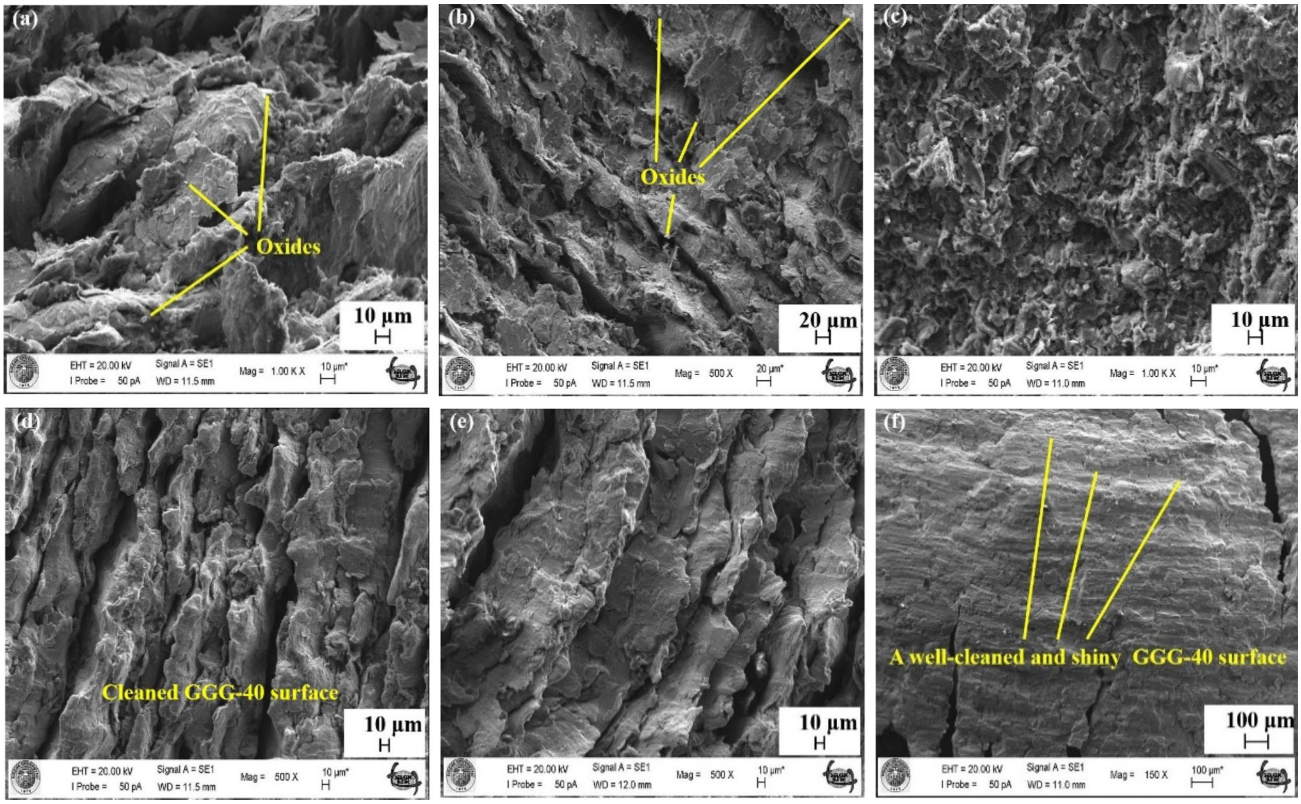


Fig. 5 SEM images of GGG-40 metallic chips **a**, **b** uncleaned; **c**, **d** 20 min and **e**, **f** 40 min

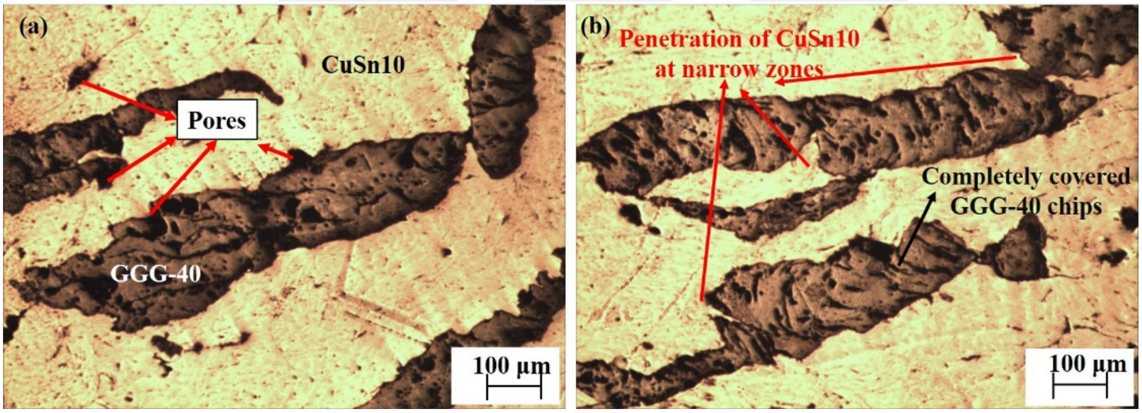


Fig. 6 Microstructures of MMC materials. **a** 60B40C specimen and **b** 90B10C specimen

efficiency of GGG-40. Also, prismatic-shaped specimens exhibit less increment than cylindrical specimens owing to the low plastic deformation ability of CuSn10 metallic chips into the prismatic die.

Porosity and Brinell hardness test results show that the results are consistent and support each other. Besides, it has been reported in some studies that hardness may decrease as the porosity increases, and hardness may increase as the porosity decreases [23, 54]. The decrease in porosity values leads to increase in Brinell hardness. Because oxides which are located at the surface of metallic chips are brittle and for this reason, they are restricted to plastic deformation. Restriction of plastic deformation prevents the coverage of GGG-40 by CuSn10 metallic chips and low quality of mechanical interlocking [54, 55]. Also, oxides between chips surface get fractured due to plastic deformation and can result in bi-film formation [56]. Bi-film is a weak region formed between two surfaces with low bonding quality, and this region acts as the trigger for pore formation. In addition, an oxide layer can curl on its own during hot pressing, forming mutual layers called bi-film structures. These bi-film structures behave like weak pores and

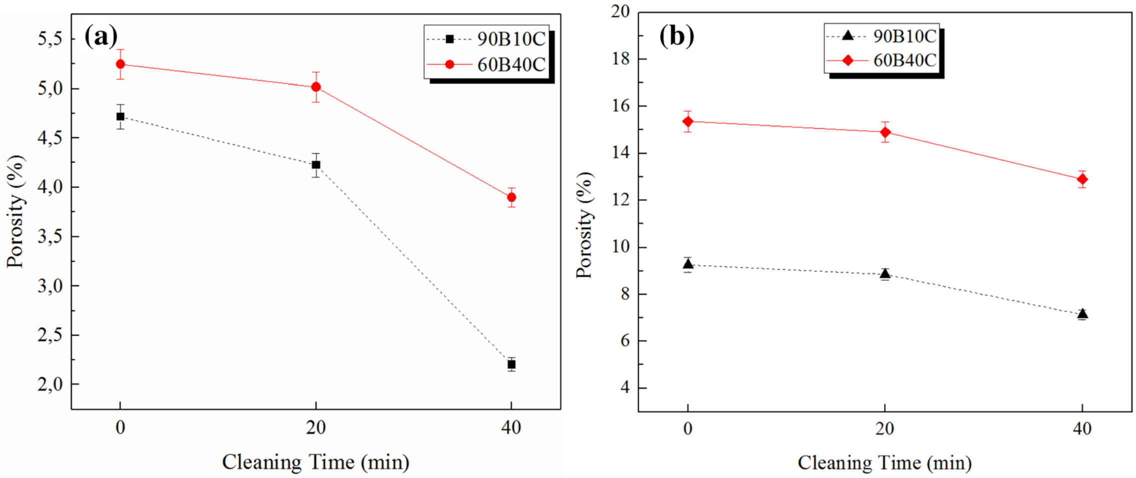


Fig. 7 Porosity test results. **a** Cylindrical specimens and **b** prismatic specimens

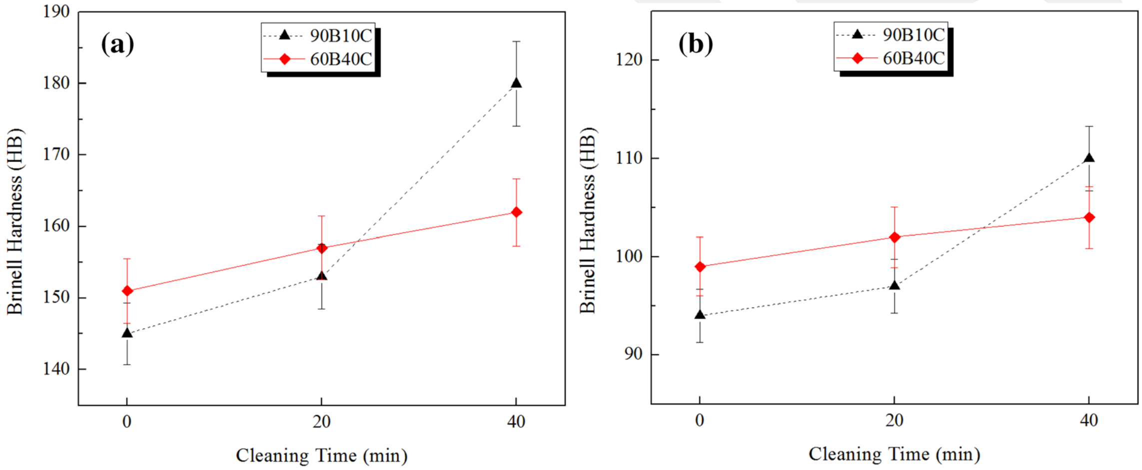


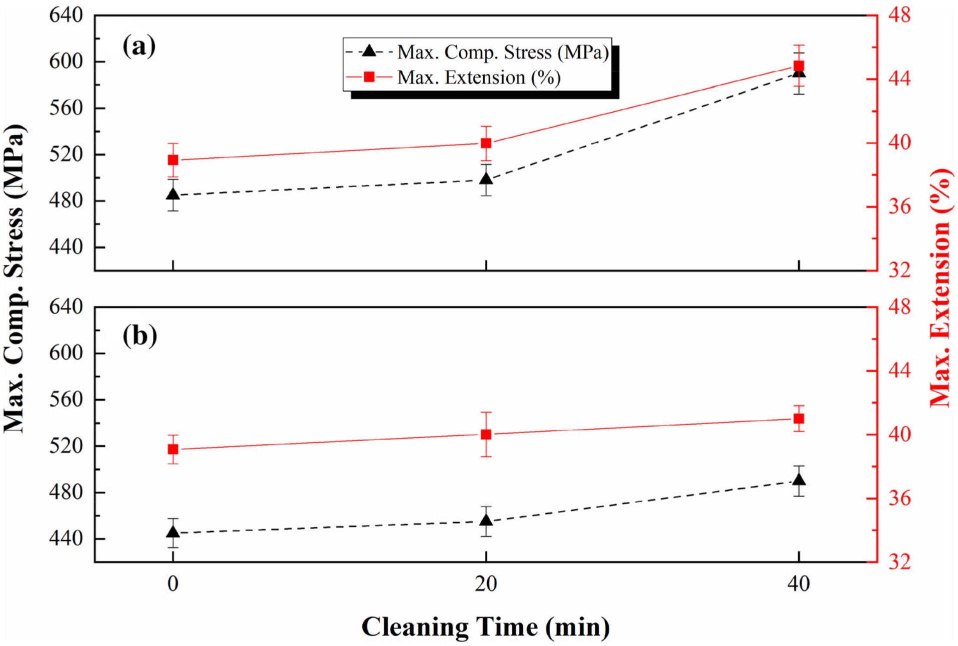
Fig. 8 Brinell hardness test results. **a** Cylindrical specimens and **b** prismatic specimens

result in a decrease in hardness and strength. In the literature, it has been reported that some air voids may remain between the bi-films [57, 58]. Because of that, the bi-film formation is an undesirable condition at hot press method. The oxides on chips that are not ultrasonically cleaned can form local non-bonded regions. This situation can be eliminated with the help of ultrasonic cleaning process. Hence, it inferred that formation of bi-film can be avoided by utilizing the ultrasonic cleaning process and it enhances the bonding efficiency of metallic chips.

3.3 Compression and Three-Point Bending Tests Results

The compression test results of cleaned and uncleaned MMC materials are shown in Fig. 9. It is observed that the ultrasonic cleaning process increases the compression strength by 4–22% for 90B10C (Fig. 9a) and by 3–7% for 60B40C (Fig. 9b) specimen based on cleaning time. In addition, it has been shown that in the case of exceeding 40 min as cleaning time, the strength of 90B10C specimen increases by 14%, while no significant changes are observed in the strength of other specimens. The increment in compression strength is related to removal of oxides and strengthening of mechanical interlocking based on ultrasonic cleaning. Bi-film effect, which is explained in the previous section is also effective in this test. In addition, it has been reported in our previous studies that the bonding mechanism occurs with mechanical interlocking [1, 12]. It is determined that the oxides do not interact chemically at the temperature at which the production is made and that there is no secondary phase or intermetallic formation in the structure. In this case, bi-films that prevent bonding mechanism can only be considered as microcracks [58].

Fig. 9 Compression test results. **a** 90B10C specimens and **b** 60B40C specimens



Pores and/or bi-films, which are located inside of the specimen, can be considered as microcracks and they can start to enlarge and advance with the application of compressive force. This movement has been identified as not intergranular but transgranular. Thus, pores stemming from bi-film effect and oxides make it easy for crack progression and this situation reduces strength and restricts the deformation ability. Therefore, as the ultrasonic cleaning time increases, the compression strength increases. The results of high CuSn10 content specimen support this evaluation owing to high cleaning efficiency of CuSn10 metallic chips. Consequently, it is clearly said that as the cleaning rate of metallic chips increases, the strength of MMC materials increases.

The three-point bending test results of MMC materials are illustrated in Fig. 10. It has been shown that flexural loads of 90B10C specimen increases by 1.5–5% and for 60B40C, specimen increases by 3–4% based on the increment in cleaning time. It has been reported in our previous studies [12] that the tensile strength of produced MMC materials is quite lower than that of compression strength. Therefore, MMC materials break under the influence of tensile stress. Hence, the increment rate of strength observed at three-point bending test is lower than the increment in compression strength. In addition, the differences between geometry of specimen may cause restriction of plastic deformation and it makes it difficult to observe the positive effects of ultrasonic cleaning process. In spite of these negative conditions, it is a significant result that there is a slight increase in strength after the three-point bending test.

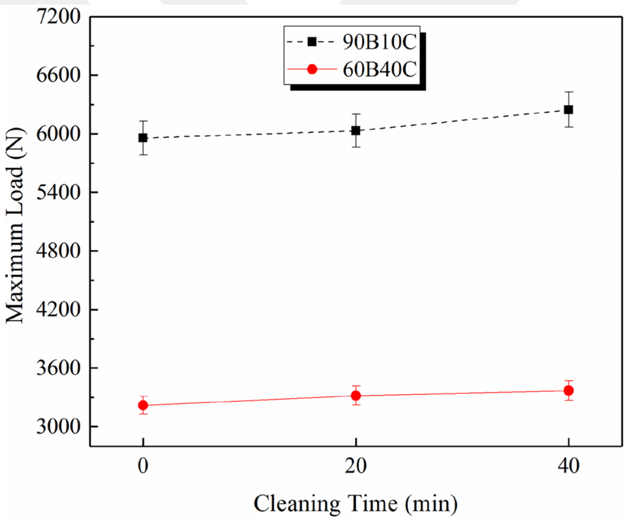


Fig. 10 Three-point bending test results

4 Conclusion

In this study, the effects of ultrasonic cleaning on the mechanical properties of cylindrical and prismatic MMC were examined. Ultrasonic cleaning process was applied at two different exposure periods. Since the cleaning efficiencies of different metallic chips were different, the effects of different mixture ratio were also investigated, and the variation in mechanical properties was evaluated. Porosity, Brinell hardness, compression and three-point bending tests were performed. The oxygen content before and after ultrasonic cleaning process was measured by means of EDX and SEM analyses. Results obtained from these tests were evaluated and listed as follows

According to EDX analysis, it was observed that CuSn10 chips could be cleaned more effectively than GGG-40 chips.

EDX analysis revealed that 40 min of cleaning time was much more effective on the cleaning process than 20 min. It was also observed that the oxides on the surface were removed in large scale, and the results were verified by SEM analysis. It was clearly seen in the SEM images that the oxides detected on the surfaces of the contaminated samples were mostly removed and the brighter surfaces were monitored.

When the results of the porosity test were examined, it was observed that there is an increment in the bonding quality of the chips depending on the increase in the cleaning time, and accordingly, the porosity values were decreased. In microstructure images, it was seen that the number of pores in the chip boundaries decreases and the GGG-40 chips cover the CuSn10 chips more effectively.

When the effect of the ultrasonic cleaning on the Brinell hardness test results was examined, it was seen that there is an increase in Brinell hardness values depending on the decrease in number of pores. Removal of the oxides on the chip surfaces resulted in better conjunction and bonding quality and thus leading to an increment in Brinell hardness values.

The compression test revealed that the increment in compression strength values was not significant as the Brinell hardness values. This could be explained by the fact that the oxides remaining in chip boundaries facilitated the progression of microcracks. An increasing number of pores which were located inside of the specimen caused easy crack progression. If the cleaning time was increased, a better bonding could be achieved in the inner regions because it was observed for all the tests, the strength was directly related to the cleaning time.

After the three-point bending test, the damage at the tensile side was observed because the samples were exposed not only to the compression stress but also to the tensile stress and the tensile strength of the samples was nominal compared to the compressive strength. Although the ultrasonic cleaning effect contributed positively to the bending strength, this contribution remained limited in comparison with other tests because of aforementioned condition and geometrical effects.

Taking all these situations into consideration, it can be said that the cleaning of the oxides on the chip surfaces is essential since oxides can result in strength deterioration and this situation can be eliminated by ultrasonic cleaning. Also, it is said that the cleaning time has a direct effect on the strength of the material and consolidation efficiency.

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