The influence of mechanical processing of chip waste from cast Al – Si – B_4C composites on the structure and properties of consolidated billets

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In order to increase the efficiency of material extraction during recycling of non-compact waste, solid-phase processing methods are currently being developed to reduce energy consumption in the manufacturing of secondary products. This study provides a comparative evaluation of the influence of high-energy ball milling of chip waste from turning ingots of AlSi12 + 10 vol.% B_4C aluminum matrix composite on changes in the structure and mechanical properties of consolidated billets compared to compacted unprepared chips and as-cast state. It was found that the samples consolidated from powder are characterized by increased strength properties in comparison with the cast composite and chips pressed composite. Specifically, the yield strength of the powder consolidated specimens increased by ~1.5 and ~2.3 times compared to the cast composite and chips pressed composite, respectively. The hardness of the cast composite was 39.35 ± 3.2 HRB, while the hardness of the samples consolidated from chips and powder was 42.75 ± 1.2 and 71.4 ± 1.5 HRB, correspondingly. The observed mechanical behavior is associated with a reduction in the sizes of the structural constituents of the matrix alloy, fragmentation of the reinforcing particles, a decrease in the porosity fraction, as well as an increase in the uniformity of particles distribution in the volume of the powder-pressed specimen. The results demonstrate the potential of solid-phase methods for processing non-compact waste in the manufacturing of products from metal matrix composites.

Key words: aluminum matrix composites, chip waste, powder metallurgy, recycling, consolidation, structure and properties

DOI: 10.17580/nfm.2023.02.07

Introduction

Metal matrix composites are a special class of heterophase materials composed of a metallic matrix and reinforcing particles of ceramic or intermetallic compounds [1]. One of the key features of metal matrix composites is the ability to purposefully control their structure and properties by varying the type, volume fraction, spatial and size distribution of reinforcing particles, chemical composition of the matrix alloy, and processing parameters for material fabrication and subsequent treatment [2–4]. Cast composite materials based on non-ferrous metals are promising for the production of high-performance components in various high-tech industries such as aerospace, automotive, special machinery, nuclear energy, oil and gas industries, and others, due to their unique properties and characteristics, including under extreme operating conditions [5, 6].

The expansion of production and application of cast metal matrix composites in industry is inextricably connected with the necessity of an effective and economical solution of the problem of recycling their scrap and waste. Repeated melting of cast composite materials can lead to the degradation of reinforcing particles due to their chemical interaction with the matrix material, which will eventually reduce the quality of products from secondary composites [7–9]. The intensity of interfacial reactions during the metallurgical processing of metal matrix composites depends on both the temperature-time parameters of the process, and the type of system under consideration, including the presence of various alloying elements in the matrix alloy. In some cases, remelting can lead to the diffusion of elements from the reinforcing components into the matrix material and changes in its chemical composition [10]. On the other hand, rational alloying of the matrix alloy can suppress the formation of undesirable interaction products at interfacial boundaries, thereby increasing the ability of metal matrix composites to be recycled by remelting [11].

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The liquid-phase processing of non-compact waste from metal matrix composites, including chips from the machining of castings and ingots, is associated with even greater technological difficulties. For aluminum alloys, it is known that the large specific surface area of the chips results in their high chemical activity, leading to significant material losses during melting processes [12]. To increase the degree of material recovery during the recycling of non-compact waste aluminum alloys, solid-state processing methods are being developed that simultaneously reduce energy costs for fabricating secondary products [13–15]. In this context, solid-state recycling processes for chip waste are being positioned as one of the ways to reduce the negative impact of production processes on the environment since they allow the production of the final product with less consumption of material and energy resources [16]. However, issues related to the solid-state recycling of non-compact waste from the machining of cast aluminum matrix composites, particularly the Al – Si – B_4C system, remain insufficiently elaborated. Furthermore, there is quite limited information about the effect of mechanical processing of chip waste from aluminum matrix composites on the structure and properties of consolidated billets, which is critically important for the development of scientifically-based technologies for producing end products.

The purpose of this work is a comparative assessment of the effect of high-energy ball milling of chip waste from turning ingots of AlSi12 + 10 vol.% B_4C aluminum matrix composite on changes in the structure and mechanical properties of consolidated blanks compared to compacted unprepared chips and as-cast state.

Materials and Methods

The initial composite material, AlSi12 + 10 vol.% B_4C, was produced by mechanical stirring of powder particles of the reinforcing phase, boron carbide F220 (principal fraction 75–63 μm), into the melt of the AlSi12 aluminum alloy using a stainless steel AISI 316 impeller. The matrix alloy was used in the as-received condition, loaded in pieces into a furnace preheated to 600–700 °C, and overheated after melting to a temperature of 850 ± 5 °C. The stirring time was 10 min at an impeller rotation speed of 300 rpm. The prepared melt was poured at a temperature of 750 ± 5 °C into a cylindrical steel mold with an inner diameter of 30 mm and a height of 100 mm. Samples were cut from the ingots for the investigation of the structure and mechanical properties.

To obtain chips, ingots were subjected to external turning on the ATPU-125 machine. For selecting the regime parameters of the turning machining, the recommendations of previous studies [17, 18] were taken into account. Turning was carried out using a feed-through cutter with a cutting part made of cubic boron nitride at a spindle rotation frequency of 3000 rpm, a cutting depth of 0.5 mm, and a feed rate of 0.1 mm/rev. The chips resulting from the turning operation were milled in a Fritsch Pulverisette 6 planetary ball mill using surface-active substances (0.5 wt.% stearic acid). A ceramic pot made of ZrO_2 and ceramic balls with a diameter of 8 mm of the same material were used for milling. The processing was carried out at a rotational speed of 400 rpm. To maintain the thermal regime, the processing was paused for 10 min every 15 min. The ratio of the weight of the chips to the weight of the grinding bodies was 1 : 15. The total processing time was 4.5 hours.

The consolidation of the composite powder obtained by milling and the initial chips, in accordance with the recommendations of previous work [19], was carried out in a mold preheated to 450 °C at a pressure of 330 MPa. The holding time under pressure was 10 min. As a result, compact cylindrical samples with a diameter of 17 mm and a height of 10 mm were obtained.

The surface morphology of the chips and the synthesized composite powder was investigated using the FEI Quanta 200 3D scanning electron microscope. The phase composition of the initial ingots, chips, powder, and samples consolidated from chips and powder was studied by *X*-ray diffraction analysis on the Bruker D8 Advance diffractometer.

The microstructure of the consolidated samples was examined using an Altami Met 1-C optical metallographic microscope in the bright field mode on unetched metallographic specimens prepared using Allied sample preparation equipment. The residual porosity of composite ingots and samples consolidated from chips and powder was determined by hydrostatic weighing.

The hardness of cast and composite samples consolidated from chips and powder was measured by HRB scale at a load of 1000 N using a Time Group TH300 hardness tester. The arithmetic mean values and the confidence intervals of the measurement results were determined.

Compression tests of cast and consolidated composite samples were performed on a Time Group WDW-100E universal testing machine at a compression rate of 0.1 s^{-1}. Cylindrical samples with a diameter of 6 mm were obtained by electrical discharge cutting on a Mitsubishi BA8 machine. The specimens were placed in the body of the mold evenly in a circle during cutting. The longitudinal axis of the samples coincided with the direction of force application during pressing. Graphite lubricant was used to reduce friction on the end faces of the samples.

Results and Discussion

Fig. 1 presents typical SEM images with different magnifications of the chips obtained after turning the ingots made of the AlSi12 + 10 vol.% B_4C aluminum matrix composite. Analysis of SEM images shows that turning ingots results in articulate-type chips (see **Fig. 1, *a***). The length of the chip fragments was ~2 mm. Cracks and discontinuities that appear due to intensive deformation of the material during cutting under conditions of high pressure and friction force can be seen on the side boundaries of the chip (see **Fig. 1, *b***). Shear bands that appear

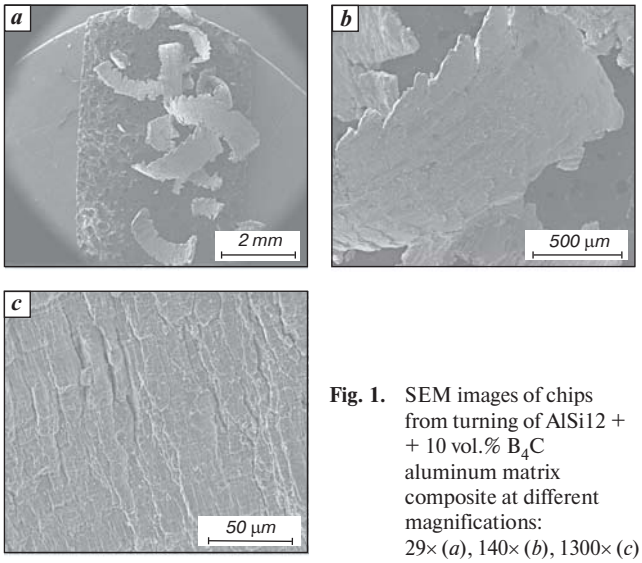

Fig. 1. SEM images of chips from turning of AlSi12 + + 10 vol.% B_4C aluminum matrix composite at different magnifications: 29× (*a*), 140× (*b*), 1300× (*c*)

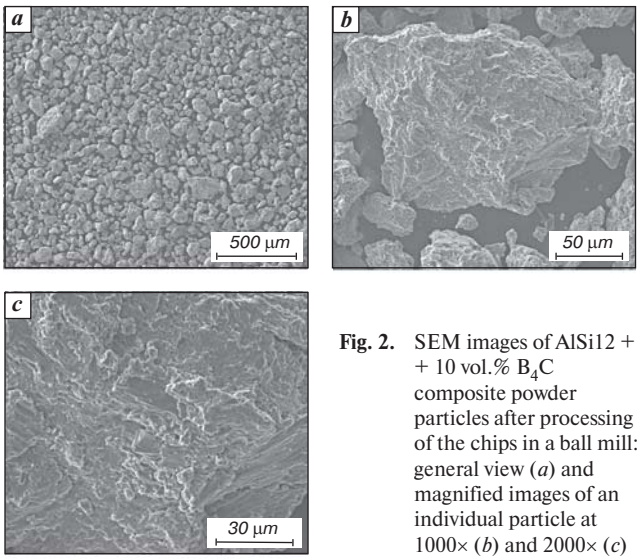

Fig. 2. SEM images of AlSi12 + + 10 vol.% B_4C composite powder particles after processing of the chips in a ball mill: general view (*a*) and magnified images of an individual particle at 1000× (*b*) and 2000× (*c*)

due to a high degree of deformation are clearly visible on the free surface of the chips (see **Fig. 1, *c***). The identified features can not only contribute to changes in the structure of the matrix alloy compared to the as-cast state but also lead to the fragmentation of the reinforcing phase.

The processing in a planetary ball mill led to a significant reduction in chip size and the formation of a powder mixture. **Fig. 2** show typical SEM images of the powder obtained after high-energy ball milling with different magnifications. SEM analysis shows that the resulting powder mixture is quite homogeneous (see **Fig. 2, *a***). The powder particles have an irregular shape, and their size does not exceed 200 μm. B_4C particles with a size of ~5–15 μm were fixed on the surface of the matrix alloy particles (see **Fig. 2, *b–c***), indicating the refinement of the reinforcing phase during processing in the planetary mill.

The typical microstructure images of the as-cast composite and samples consolidated from chips and powders obtained by optical microscopy are shown in **Fig. 3**. Metallographic analysis reveals that the microstructure of the cast composite material (see **Fig. 3, *a–b***) consists of an AlSi12 matrix alloy with α-solid solution (lighter areas with dendritic morphology), eutectic (α + Si) with distributed B_4C microparticles (dark gray inclusions) having a characteristic splinter shape. It can be observed that the cast composite shows a tendency for agglomeration of B_4C reinforcing particles, which is a common problem for composites obtained by metallurgical methods [20]. In addition, the agglomeration of B_4C particles has resulted in the presence of pores caused by air entrapment during mechanical stirring of the melt. In total, this may reduce the effectiveness of strengthening the cast composite with ceramic particles [21, 22].

The microstructure of the sample consolidated from the chips had a different appearance (see **Fig. 3, *c–d***). The areas belonging to the α-solid solution had a non-dendritic

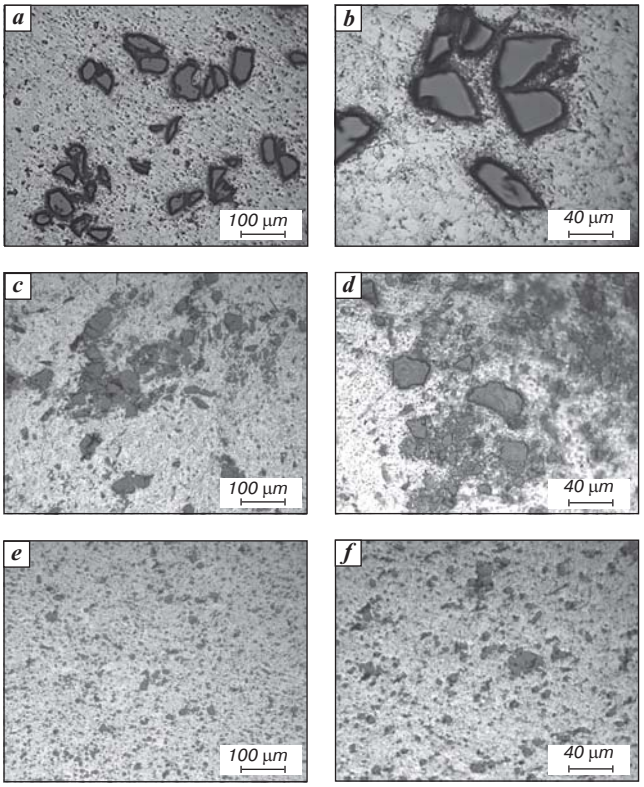

Fig. 3. Typical microstructure images of AlSi12 + 10 vol.% B_4C aluminum matrix composite at different magnifications for as-cast (*a*, *b*), consolidated from chips (*c*, *d*), and pressed from powder (*e*, *f*) samples

morphology. The change in structure was apparently caused by the thermo-deformation impact on the machined material during turning and subsequent compacting. Along with large reinforcing particles, a sufficient number of smaller ones, less than 15 μm in size, can be observed. Considering that the presence of small reinforcing phase particles was not detected in the cast samples,

their appearance in the chip sample, as expected, is caused by partial crushing of the reinforcing phase during cutting. At the same time, relatively large agglomerates were present in the structure of the sample consolidated from the chips, similar to those in the cast sample.

The microstructure of the sample consolidated from powder differed significantly from both the cast and chip-pressed samples (see **Fig. 3, *e–f***). In particular, the uniformity of the reinforcing particle distribution was significantly improved, as confirmed by the absence of B_4C agglomerates in the microstructure images. Judging by the images, the average size of the B_4C reinforcing particles and the porosity of the material were significantly reduced.

Fig. 4 shows the *X*-ray diffraction patterns of cast, consolidated from chips, and pressed from powder composite specimens. A comparative analysis of the diffraction patterns shows their qualitatively similar character.

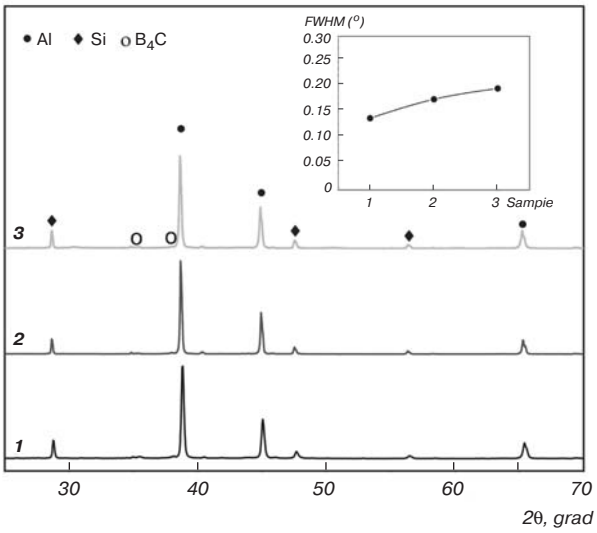

Fig. 4. The results of *X*-ray diffraction analysis and data on the FWHM of the Al (111) peak (inset):
1 – cast sample; *2* – chip-pressed sample; *3* – powder-pressed sample

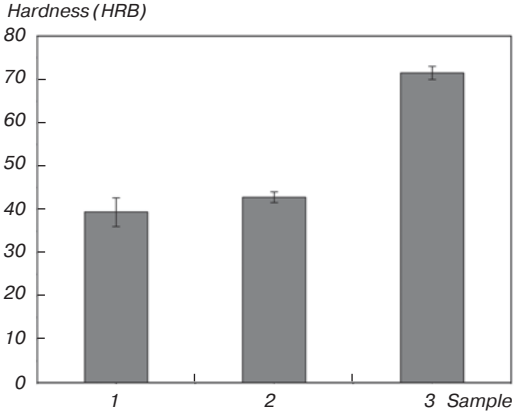

Fig. 5. Hardness of AlSi12 + 10 vol.% B_4C aluminum matrix composite:
1 – as-cast sample; *2* – chip-pressed sample; *3* – powder-pressed sample

The samples exhibit strongly pronounced Bragg diffraction peaks corresponding to aluminum and free silicon. In addition, weakly intense peaks of the B_4C reinforcing phase were observed. The analysis of the diffraction patterns does not show the presence of peaks (halos) of the native Al_2O_3 phase, which should inevitably form in both the chip and powder samples.

The inset in **Fig. 4** shows data characterizing the full width at half maximum (FWHM) of the aluminum (111) peak for the investigated samples. Analyzing the variation of this characteristic, it is possible to qualitatively estimate the change in the size of the structural components of the matrix alloy. It can be seen that the FWHM value for the samples from chips and powder is higher than for the ingot. This is due to the surface and volumetric cold-work hardening typical for plastic deformation processes occurring during turning and high-energy ball milling, which lead to a decrease in the size of the structural components of the matrix material.

The residual porosity of the as-cast sample was 2.7 ± 0.74%. Meanwhile, the average residual porosity for the sample consolidated from chips was 5.4 ± 0.32%. For samples consolidated from powder, an experimentally measured density exceeding the theoretically calculated one by the rule of mixtures was observed. The average density of the powder-pressed samples was 2.67 g/cm^3, corresponding to a relative density of 101.1 ± 0.19%. A reasonable explanation for this circumstance is the presence of native Al_2O_3 in the powder samples. Assuming that the real relative density of the pressed samples is 99%, the fraction of native Al_2O_3 calculated by the rule of mixtures does not exceed 1.65 vol.%. Apparently, this value is below the detection limit of *X*-ray diffraction analysis.

The hardness of samples consolidated by hot pressing was expectedly higher than that of cast samples **(Fig. 5)**. For instance, the hardness of the ingot was 39.35 ± 3.2 HRB, while the hardness of the samples consolidated from chips and powder was 42.75 ± 1.2 and 71.4 ± 1.5 HRB, respectively. Therefore, the chip and powder samples had hardness values that were 8% and 80% higher than the cast sample, correspondingly. Considering the FWHM data characterizing the change in the size of the structural components of the matrix alloy, as well as the optical microscopy data, it can be suggested that the increase in the hardness of the samples obtained from machining waste is related to a decrease in the size of the structural components and reinforcing phase, an increase in the macro-density of pressing, and an increase in the homogeneity of the distribution of B_4C reinforcing particles in the case of the powder-pressed sample.

Fig. 6 shows the deformation curves under compression conditions for as-cast, chip-consolidated, and powder-pressed specimens.

Compression testing of the samples reveals that the cast composite materials exhibit a higher level of ductility compared to powder-consolidated samples. The strain to failure of the latter does not exceed 10%, whereas the cast

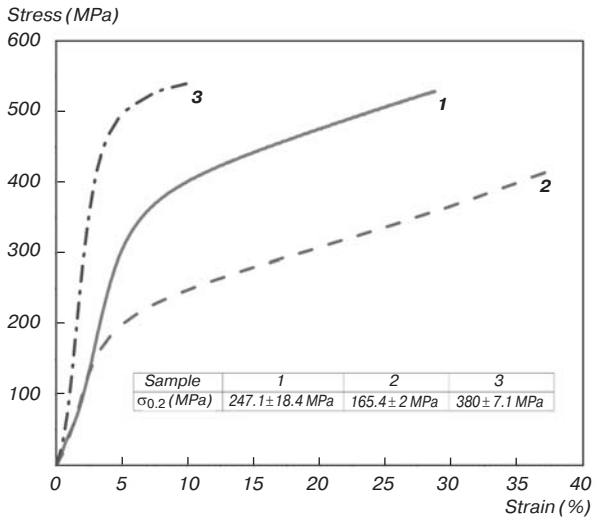

Fig. 6. Typical compression curves:
1 — as-cast sample; *2* — chip-pressed sample; *3* — powder-pressed sample

composite material undergoes a strain of 25% without failure. At the same time, the composite pressed from chips is also characterized by high ductility. Nevertheless, the samples consolidated from powder exhibit increased mechanical properties compared to the as-cast and chip-pressed samples of AlSi12 + 10 vol.% B_4C aluminum matrix composite. Specifically, the yield strength increases by ~1.5 and ~2.3 times compared to the as-cast and chip-pressed samples, respectively. For powder-consolidated samples, the yield strength was 380 ± 7.1 MPa, while for the cast composite, this parameter was 247.1 ± ± 18.4 MPa. A decrease in yield strength to 165.4 ± 2 MPa is observed for the chip-pressed sample. The strength properties of the powder-pressed composite increase due to an improvement in the uniformity of distribution and a decrease in the size of the reinforcing particles during high-energy ball milling. Apparently, such a change in the mechanical behavior may be due to the fact that during deformation, large reinforcing particles are less effective due to the wider interfacial area with the matrix and higher concentration of pressure, as well as the preferential concentration of stress during loading of the sample at the places of clustering of reinforcing particles.

Overall, the obtained results confirm that the chips generated during the mechanical processing of ingots from metal matrix composites can be recycled as an initial material for the production of secondary composites via powder metallurgy methods. However, to ensure the enhanced mechanical properties of the recycled composites, a necessary stage in the technological process is the pre-processing of the chip waste in a planetary ball mill to obtain a powder material.

Conclusions

The results indicate that the pre-processing of chips obtained from machining of AlSi12 + 10 vol.% B_4C cast aluminum matrix composites in a high-energy ball mill significantly enhances the mechanical properties of the composites after solid-state recycling. This is attributed to the reduction in the size of structural constituents of the matrix alloy and the fragmentation of the reinforcing particles, decrease in porosity, and an increase in the homogeneity of particles distribution within the sample obtained by powder pressing. The yield strength of the consolidated powder samples increases by ~1.5 and ~2.3 times compared to as-cast and chip-pressed samples, respectively. Overall, the use of powder metallurgy methods for producing secondary composites from waste generated after machining of castings and ingots can contribute to the enhancement of economic efficiency of chip recycling processes.

Acknowledgments

This research was funded by the Russian Science Foundation (Project № 21-79-10432, https://rscf.ru/project/21-79-10432/). The study was carried out using the equipment of the interregional multispecialty and interdisciplinary center for the collective usage of promising and competitive technologies in the areas of development and application in industry/mechanical engineering of domestic achievements in the field of nanotechnology (Agreement No. 075-15-2021-692 of August 5, 2021).

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