

## Article

# Effect of Magnesium Matrix Grain Refinement Induced by Plastic Deformation in a Composite with Short Carbon Fibers

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**Abstract:** The magnesium matrix composite reinforced with 3 vol. % of short carbon fibers (Csf), fabricated, under industrial conditions, by the stir casting method, was applied to obtain composite bars by two extrusion methods: the novel method of cold severe plastic deformation with a forward-backward rotating die (KoBo) and conventional extrusion at 400 °C. The effect of Mg( $\alpha$ ) grain refining, as well as fibers behavior and phenomenon at the fiber-matrix interface, was examined by optical microscopy, scanning electron microscopy with energy dispersive spectroscopy and scanning-transmission electron microscopy methods. The Mg( $\alpha$ ) grain quantitative characteristics revealed a decrease of the equivalent diameter from  $219 \pm 76 \mu\text{m}$  (as-cast) to  $24 \pm 10 \mu\text{m}$  and  $0.89 \pm 0.35 \mu\text{m}$  (the hot-extruded and KoBo-processed, respectively). In addition, due to the KoBo application, except for the Csf orientation that was parallel to the extrusion direction, an effect of fibers fragmentation on the length of few Csf diameters was detected. No significant changes in the Csf-matrix interface (besides those between new carbon surfaces) formed by fibers fragmentation, and the matrix created by extrusion were detected. A comparison of the mechanical properties of the Mg-Csf composite showed that the KoBo method ensured a spectacular increase in strength and plasticity.

**Keywords:** magnesium matrix composite; short carbon fibers; hot extrusion; several plastic deformation

## 1. Introduction

The control and significant refining of the metal matrix grain size in composites processed by liquid technologies induce some limits because during the process of fabrication, both the grain size and the shape are mainly determined by primary crystallization. Additionally, it should be noted that the growth of primary crystals may cause a cellular distribution of the particles or short fibers in the composite [1–4]. Therefore, in some technologies, including magnesium composite fabrication, increase the homogeneity, as well as close the micropores formed both in casting and powder metallurgy processes, plastic deformation is applied as a step of the technological procedure [5–7]. In conventional processes, to avoid microcracks generation in the matrix and reinforcing phases, as well as at the matrix-reinforcement interface, deformation by hot plastic working (for instance [2,5,7], hot extrusion) is employed. However, an increase in the processing temperature causes metal matrix recovery and recrystallization, and in practice, the decrease of the metal grain size occurs but is limited.

For the last few years, different methods of severe plastic deformation (SPD) have been successfully applied to light metal alloys without preheating, and a spectacular increase in the mechanical properties due to refining the grain size to a submicroscale, and even nanoscale has been obtained [8–12]. One of them is the KoBo method (i.e., cold extrusion with a forward-backward rotating die), which is used for different metallic material processes.

This study seeks to prove how plastic deformation methods can be effective for a magnesium matrix composite with short carbon fibers (Csf) in terms of the evolution of the microstructure, properties

and potential fabrication under industrial conditions. The KoBo method was earlier proposed in the laboratory research of composites with aluminum [13] and silver matrices [14] with ceramic micro-sized particles, but for the magnesium composites, the technology is under consideration for the first time. In our study, a cast composite with Csf was applied as the initial material, and a noncommercial magnesium alloy with zinc (Zn), zirconium (Zr) and rare earth elements (REE) was used as the matrix. The composite was fabricated by the stir casting method under industrial conditions. Then cold extrusion by the KoBo method was employed, as well as well-known conventional hot extrusion for comparison. In this study, the mechanical properties of Mg-Csf composite bars, obtained by two extrusion methods, were determined, and microstructure examinations were carried out to explain the reason for the spectacular increase induced by extrusion processes. Changes in the size of the Mg( $\alpha$ ) grains, as well as the behavior of the short fibers, and the stability of bonding between the fiber and the matrix that was previously formed in initial cast material were analyzed. The results of the experiment are indications of the perspective for commercial fabrication of Mg-Csf composite bars by the extrusion methods.

## 2. Materials and Methods

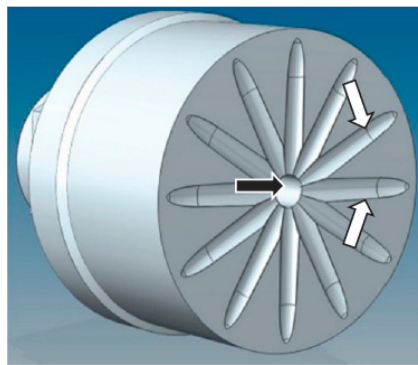
The initial composite material with a magnesium matrix alloyed with 2.4 wt. % Zn, 0.63 wt. % Zr, 0.6 wt. % REE was fabricated from a gravity cast metal-fiber suspension [4,15], and Csf (Tenax STS 5631 1600 tex), in the form of 3D granules and length of 40–250  $\mu\text{m}$ , were employed. The Csf content in the composite was approximately 3 vol. %, and it was the limit value for the applied industrial conditions, where the mass of one melt was 40 kg. The processing of the gravity cast composite samples, 40 mm in diameter and 50 mm high, by conventional hot extrusion at 400 °C was carried out after preheating at 400 °C for 2 h, and 12 mm diameter bars were obtained. It means a degree of processing (ratio of cross-section area before extrusion to cross-section area after extrusion),  $\lambda = 11.11$ . Such parameters of hot extrusion were selected based on earlier own experiments focused on magnesium. Further experiments focused on composite bar diameter reduction by hot extrusion were unsuccessful because in the macro- and micro-scales, delamination was observed, and its effect of structural degradation was detected both on the inside of the composite bar and at its surface.

In the KoBo method, the samples without preheating, sized as applied for hot extrusion, were reversibly twisted ( $\pm 8^\circ$ ) just before entering the die, where a cross section was reduced. In Figure 1, a scheme of periodically rotated die applied for composite bar processing by KoBo extrusion is presented, and the grooves characteristic for this method located on the die front are demonstrated. At first, the frequency of 5 Hz was applied and then a lower frequency, until a force of 1 MN and a punch displacement rate of 0.2 mm/s were obtained. Finally, 6 mm diameter bars were obtained—that is,  $\lambda = 44.4$ . It must be mentioned that in KoBo extrusion, a temperature of processed material increases due to friction with the die material, as well as internal friction induced by intense several plastic deformation. Because a direct temperature measurement of the extruded material was difficult to obtain (due to a closed space), the temperature of the composite was determined with a thermographic camera just behind the KoBo die and was estimated as approximately 200 °C.

The composite bars fabricated in experiments by two extrusion methods are shown in Figure 2. After hot extrusion, the composite bars possessed the surface of dark gray matte color (Figure 2a). The composite bars were glossy and metallic, with very fine regular relief, after KoBo extrusion application (Figure 2b). The observed difference in surface character indicated that a very short temperature increase of the magnesium base composite in KoBo extrusion did not induce strong oxidation effects opposite to the hot extrusion.

A tensile test (Zwick/Roell Z100, Zwick/Roell, Ulm, Germany) and microhardness measurements (HV0.2, Zwick/Roell, Ulm, Germany) were employed for characterization of the composite's properties. Microstructure examinations of polished and fractured samples were carried out using an optical microscope (OM, Olympus GX51, Olympus, Tokyo, Japan) and a scanning electron microscope (SEM, Hitachi S 4200, Hitachi Group, Tokyo, Japan). The thin foil prepared by the "lift out" method with a focus

ion beam was examined with a scanning-transmission electron microscope (STEM, Hitachi HD-2300, Hitachi Group, Tokyo, Japan). The grain size was characterized by the equivalent diameter ( $d_2$ ), estimated with the use of computer analysis of the microstructure images by the Metilo program [16]. To calculate the equivalent diameter, the OM images of as-cast and hot-extruded composites were applied, while the STEM images of the KoBo extruded material. To exclude the influence of potential grain orientation on the measurement results, the samples transversed to the extrusion orientation were examined.



**Figure 1.** A scheme of the periodically rotated die with marked die opening (black arrow) and grooves (white arrows) applied to Mg-Csf composite bar cold working by KoBo-extrusion.



(a)



(b)

**Figure 2.** A view of Mg-Csf composite bars after hot extrusion at 400 °C (a) and KoBo-extrusion (b).

### 3. Results and Discussion

The mechanical properties measured for the Mg-Csf composite as-cast and extruded by the two methods are presented in Table 1. The obtained results show that the composite deformed by the KoBo method demonstrated the highest hardness. In both extruded materials, the hardness measured at a perpendicular orientation to the extrusion direction at the surface was higher than at a parallel orientation, which is an indication of the transformation of the initial cast material microstructure from an isotropic into an anisotropic material. The yield strength, ultimate tensile strength and elongation (Table 1), independent of the extrusion method, were also higher. These results confirmed the well-known benefits of plastic deformation applied to close the primary micropores, both in the

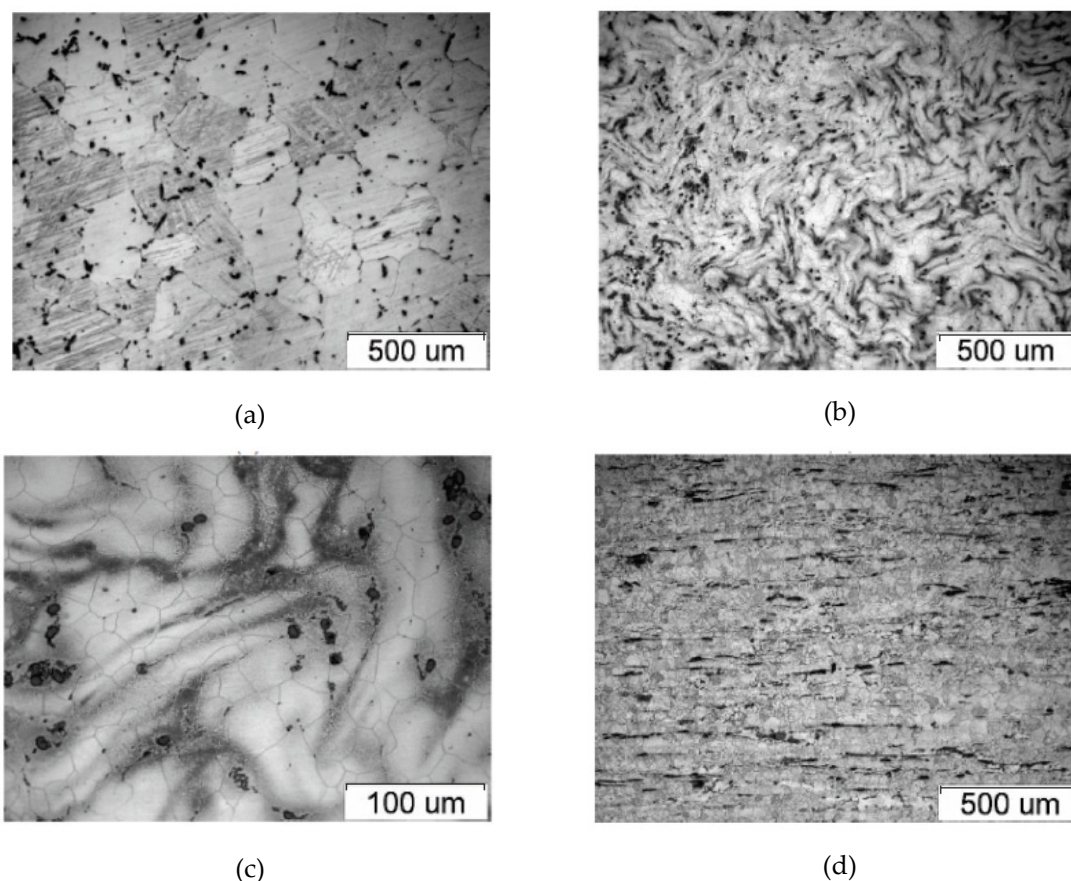
matrix and in the reinforcing phase-matrix interface. However, a great increase in all the measured properties indicates the microstructure transformation as the crucial source.

**Table 1.** Mechanical properties and equivalent Mg( $\alpha$ ) grain diameter of Mg-Csf composite processed by different technologies.

Technology	HV0.2	$R_{0.2}$ , MPa	$R_m$ , MPa	A, %	$d_2$ , $\mu\text{m}$
Gravity casting	$47 \pm 2$	$50 \pm 8$	$70 \pm 11$	$1.4 \pm 0.3$	$219 \pm 76$
Hot extrusion	$^{\perp}56 \pm 1$ , $\parallel 52 \pm 2$	$160 \pm 6$	$230 \pm 9$	$10 \pm 1.5$	$24 \pm 10$
KoBo extrusion	$^{\perp}78 \pm 4$ , $\parallel 71 \pm 2$	$270 \pm 12$	$301 \pm 11$	$14 \pm 2$	$0.89 \pm 0.35$

<sup>⊥</sup> surface perpendicular oriented to the extrusion direction;  $\parallel$  surface parallel oriented to the extrusion direction.

Examples of the performed microstructure examination are shown in Figures 3–9. In the as-cast composite, the matrix grains of equivalent diameter  $d_2 = 219 \pm 76 \mu\text{m}$  (Table 1), and the characteristic cellular distribution of the Csf were visible (Figure 3a). Hot extrusion caused an absence of that effect (Figure 3b–d), but it induced fiber orientation, almost parallel to the extrusion direction, as well as a decrease of  $d_2$  of one order of magnitude ( $24 \pm 10 \mu\text{m}$ ). Additionally, the absence of grain orientation in the longitudinal cross sections was revealed (Figure 3d), which suggests recovery and recrystallization.

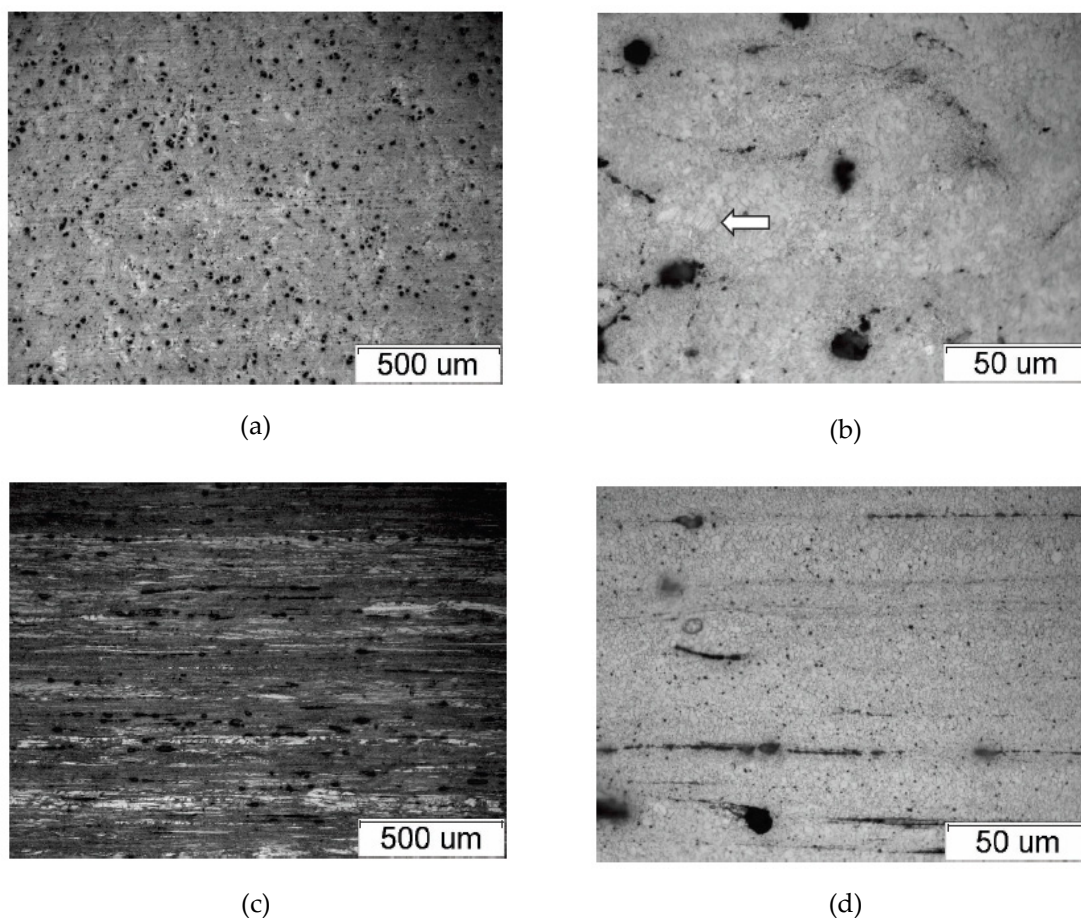


**Figure 3.** OM micrographs of Mg-Csf composite sections: (a) as-cast and hot-extruded, (b,c) transverse and (d) longitudinal.

In the case of the KoBo-processed composite (Figure 4), the fiber distribution was similar to that in the hot-extruded composite. There were no fiber cells, but the orientation was almost parallel to the working direction. That orientation was also specific for the phases insoluble in Mg( $\alpha$ ) as



oxides, intermetallics and zirconium because they formed the bands. The Mg( $\alpha$ ) grains were ultra-fine,  $d_2 = 0.89 \pm 0.35 \mu\text{m}$  (Table 1), but incidentally, non-recrystallized primary grains were detected in the transverse section (Figure 4b). It is characteristic that despite the material was not preheated during processing by the KoBo method, the refined grains were equiaxed in the longitudinal cross sections. A similar effect, but for metal alloys, has been observed and described in many studies [9–11]. The results of this study indicate that the differences in bar hardness dependent on the orientation were not caused by elongated Mg( $\alpha$ ) grains but by the orientation of the fibers and bands formed by intermetallics, oxides and zirconium. The examinations of the KoBo-processed composite by STEM revealed effects of recrystallization and characteristic straight and high angle grain boundaries, tangles of dislocations and single dislocations (Figure 9), but first of all, they confirmed that the matrix grains achieved submicrosizes, and even nanosizes. It must be mentioned that the microstructure characterization of the fiber-matrix interphase zone by STEM was not possible because the bonding between components in thin foil prepared with an ion beam was very weak.

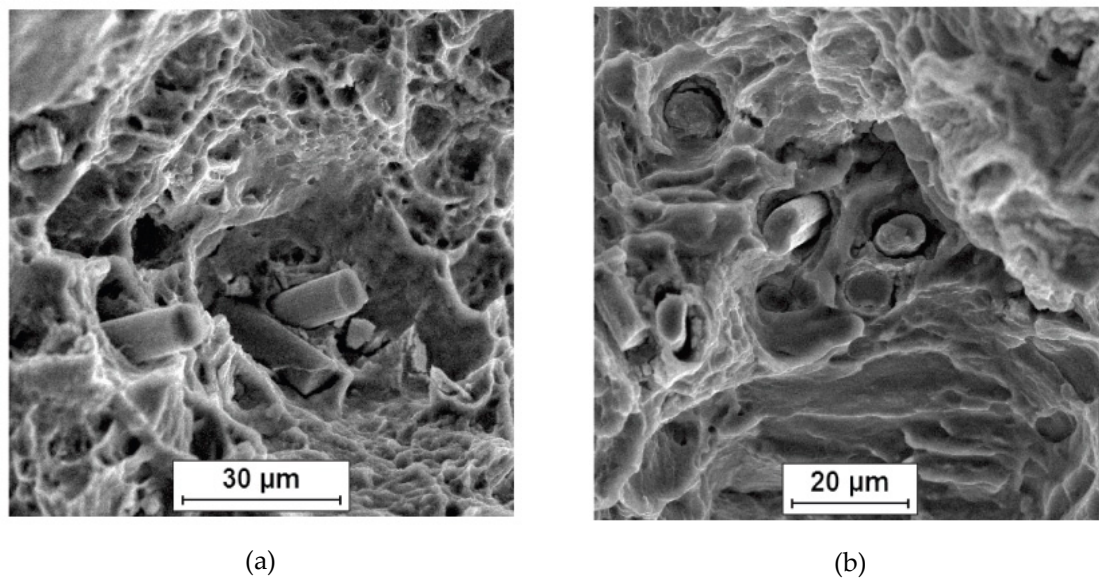


**Figure 4.** OM micrographs of KoBo-extruded Mg-Csf composite sections: (a,b) transverse and (c,d) longitudinal, white arrow indicates non-recrystallized primary grains.

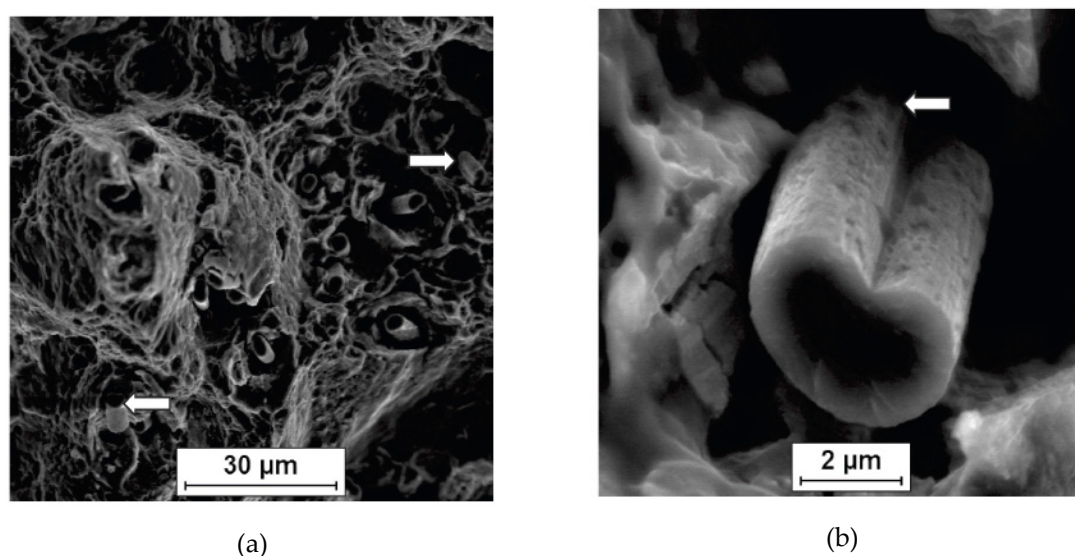
Examples of fractured surfaces after a tensile strength test are shown in Figure 5, and they reveal a difference in the matrix decohesion character. The composite samples extruded by the KoBo method exhibited ductile fracture with very fine voids (Figure 5a), while the fracture of the hot-extruded samples was of the pseudo-ductile type (Figure 5b), and that explains why the former exhibits better plasticity, despite the evidently finer grains (Table 1).

Significantly higher mechanical properties, as well as the possibility of profile reduction (higher  $\lambda$  value), caused that the studies were focused on effects induced in the Mg-Csf composite by KoBo processing. The SEM analysis of the Csf's behavior in the KoBo process revealed their fragmentation

occurring perpendicularly to the longitudinal fiber axis (Figures 6 and 7) and the final length of a single fiber was at a level of few fiber diameters. At the fractured surface, the reinforcement pulling-out that is characteristic of fibrous composites was not intense (Figure 6), but single fibers, very short and un-bonded with the matrix were noted. Additionally, at the cross-sectioned polished samples, the microvoids at the fiber-matrix interface were identified (Figure 7b) in some characteristic areas. They were located at the flat fibers ends and were probably induced by the intense movement during the SPD process of the magnesium and carbon fibers because they exhibited significantly different plasticity. Observations of the fractured and polished cross sections suggest that the bonding between the matrix and a new free carbon fiber surface formed because the fragmentation was very weak, probably of the adhesion type. The time of interaction between components in the KoBo process is very short and the temperature is relatively low; therefore, there is no condition for the formation of a new diffusive bond.

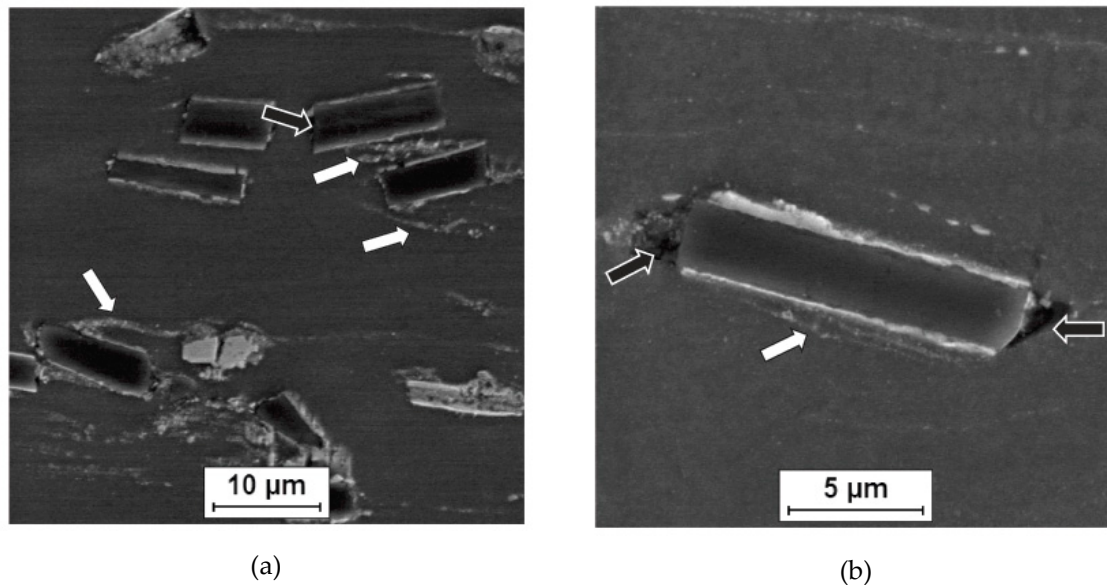


**Figure 5.** SEM micrographs of Mg-Csf composite fractures demonstrated differences in character of matrix decohesion: (a) KoBo-extruded, (b) hot-extruded.



**Figure 6.** SEM micrographs of Mg-Csf composite KoBo-extruded after strength test demonstrated effect of carbon fibers fragmentation. (a) very short fibers in pseudo-ductile matrix, (b) single fractured fiber.

Figures 7 and 8 exhibit that, unlike at the flat fibers ends, the microstructure of the interface previously formed by the casting process was not essentially destroyed at the cylindrical surfaces of carbon fibers. A thin, continuous layer enriched in oxygen and alloying elements was detected (Figure 8), which is in good agreement with earlier experiments focused on stir-cast Mg-C composites [4,15].



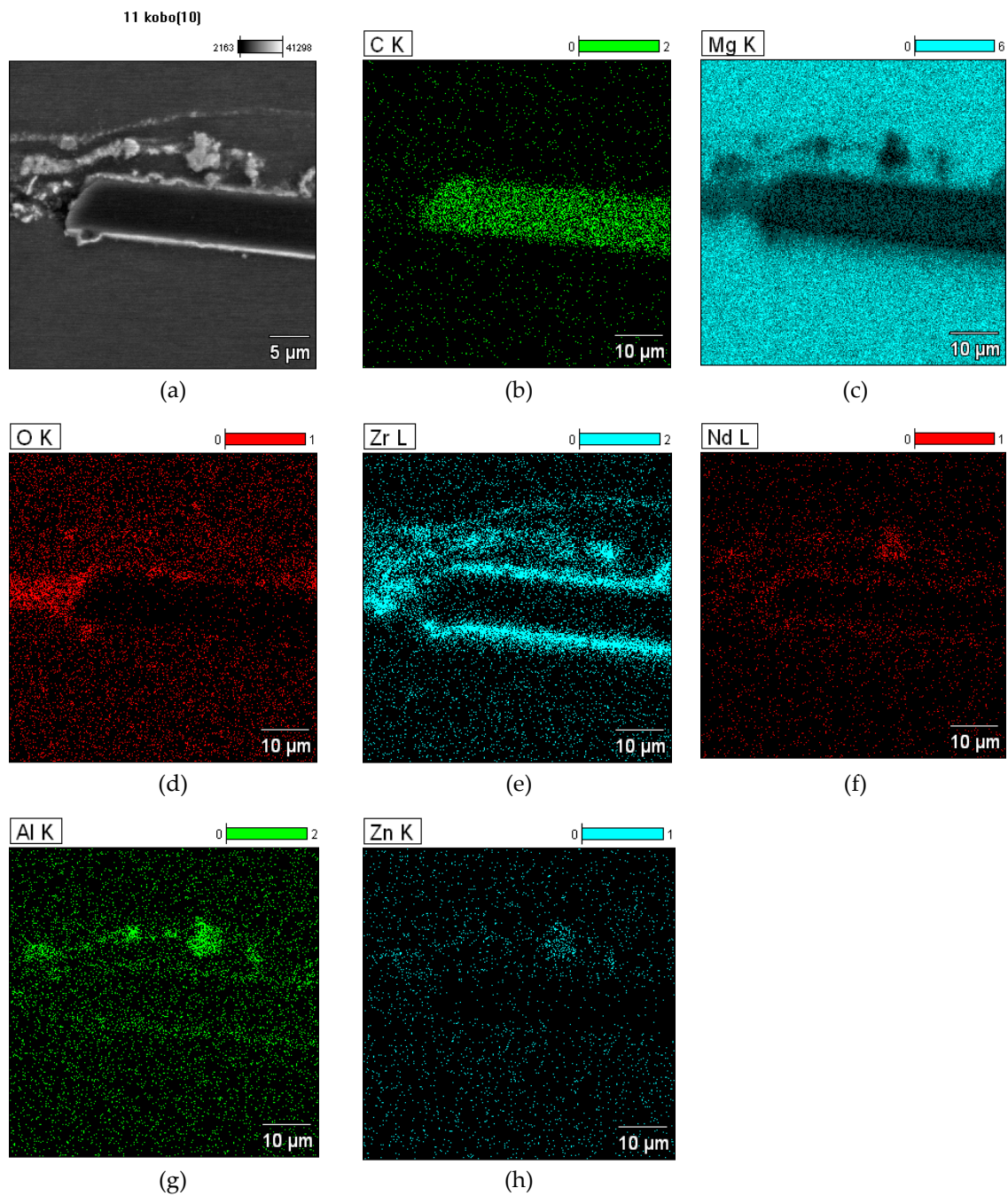
**Figure 7.** SEM micrographs of Mg-Csf composite KoBo-extruded demonstrated: (a) accumulation of intermetallics and oxides bands around (white arrows), (b) discontinuities formed between matrix and fiber (black arrows).

The results of the SEM examination presented in Figures 7 and 8 reveal light and very fine phases in the magnesium matrix, with their characteristic distribution being a consequence of the applied technological procedure. They demonstrated a layered arrangement and orientation closed to the extrusion direction, but near the stiff carbon fibers, their accumulation was observed.

With further mechanical properties increase, due to the effect of fiber fragmentation, and the absence, in KoBo extrusion conditions, of the formation of a new strong bond between fractured in situ carbon fibers and the magnesium matrix, there is no reason for further increase of the short fibers volume fraction or the continuous fiber application. However, the obtained high mechanical properties and the results of the microstructure examinations induce a question about the function of 3 vol. % of Csf with  $d = 8 \mu\text{m}$  and length  $l = 2 \div 10 \times d$  dispersed in the matrix with the  $1 \mu\text{m}$  sized grains, with regards to the composite material application. It seems that the main role of Csf in KoBo-manufactured bars can be as that of fine carbon particles, increasing the wear resistance and tribological properties of gravity- or pressure die-cast composites due to solid lubricant formation [4,17]. Moreover, as opposed to the alternative carbon additions, nanotubes [18,19] or graphene [20], proposed in the literature for the improvement of the tribological properties of light metals, Csfs are significantly cheaper, more convenient and safer for industrial use.

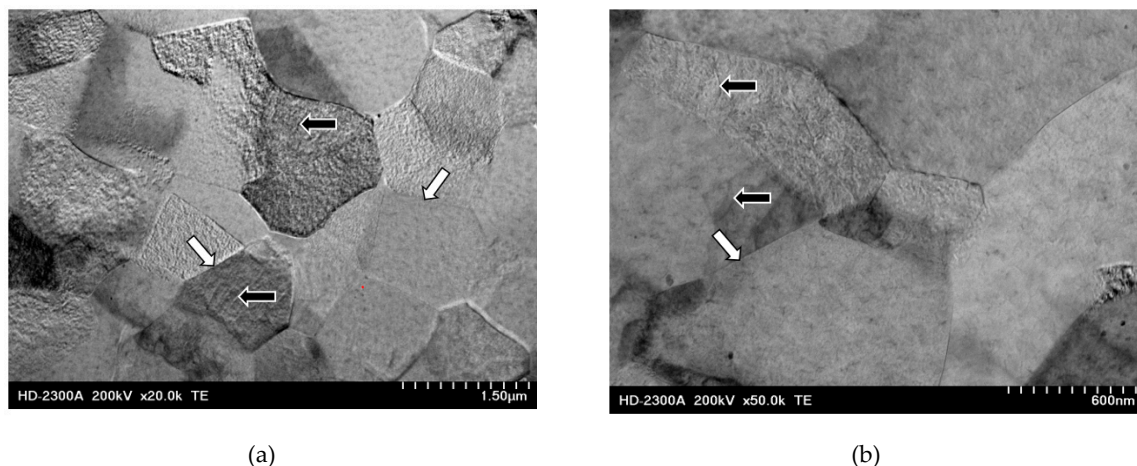
The presented experiments show the possibilities of Mg-Csf composite bars fabrication with the KoBo method, ensuring the glossy surface of the final product and very high mechanical properties due to submicrosized grains of Mg( $\alpha$ ). In comparison with conventional hot extrusion technology, the KoBo method seems to be better for the final magnesium base composite products due to better properties and appearance, a higher degree of available cross-section reduction and less self-ignition risk. Due to fibers fragmentation, the main problem that needs further examination is the proper size and shape of the initial microsized reinforcing phase that can be applied in the fabrication of magnesium matrix composite ingots.





**Figure 8.** SEM micrograph (a) and elemental mapping (b–h) of Mg-CsF composite processed by stir casting and then cold extruded by KoBo method.





**Figure 9.** STEM micrographs KoBo extruded Mg-Csf composite demonstrated effect of matrix refining; straight grain boundaries (white arrows), dislocations (black arrows).

#### 4. Conclusions

In the presented study, the possibilities of composite bars fabrication from the casts of a magnesium matrix Csf composite were analyzed with regards to microstructure modification and property improvement due to plastic deformation. For this purpose, two different extrusion methods were applied, a novel extrusion method without preheating called the KoBo method, which belongs to the group of SPD methods, and the conventional hot extrusion for comparison. After the examination of mechanical properties and microstructure characterization, the following can be concluded from this study:

- The degree of composite processing ( $\lambda$ ) that is guaranteed to obtain undefective bars was evidently higher for the KoBo method than for plastic working by hot extrusion. The KoBo extrusion led to fabrication from the same initial material, the bars of glossy metallic color with very fine regular relief, while the bars processed by hot extrusion were of dark gray matte color due to intense oxidation effects.
- The KoBo cold extrusion induced a spectacular increase in the strength and plasticity of the Mg-Csf composite in comparison with the as-cast and hot-extruded material.
- Independent of the applied extrusion method, the Mg( $\alpha$ ) grains were equiaxed and well-refined. After the hot extrusion of the Mg-Csf composite at 400 °C, the matrix grain size was reduced by one order of magnitude in comparison to those in the as-cast composite. The KoBo cold extrusion caused the formation of ultrafine matrix grains, which were two orders of magnitude smaller than those in the as-cast composite and one order smaller than those of the hot-extruded composite. Both types of extrusion induced an orientation of composite microstructure similar to the extrusion direction that contained fibers, as well as finer phases, insoluble in Mg( $\alpha$ ) as intermetallics, oxides and zirconium, which formed bands.
- An intensive carbon fiber fragmentation perpendicular to the main fiber axis occurred in the KoBo extrusion. That process generates, in carbon reinforcement, a new clean area weakly connected with the matrix and entails a decrease of fibers reinforcing effect on composite properties.
- A very high mechanical properties increase of the Mg-Csf composite bars processed by KoBo, in comparison with the cast material, can be explained by the refining of the matrix grains. That effect indicates plastic working without preheating as a very efficient technology both with respect to the fabrication of different-sized composite profiles as well as the significant improvement of properties, which is unavailable by casting methods.

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**Conflicts of Interest:** The authors declare no conflict of interest.

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