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Disorders of magnesium alloy injection molding

ABSTRACT

The feed problems in the injection molding of different magnesium alloys were investigated and explained. The examinations carried out with magnesium alloys AZ91D, ZC63 and MRI230D have shown that in addition to the known causes, other factors can also affect the draw-in disturbance. Those are following: a contaminated granulate, the unevenness and size of fine particles, a low-melting eutectic and the bevels that are used to reinforce magnesium alloys. The causes of disturbance factors that occur were analyzed and solutions were developed to eliminate and avoid them

Keywords: injection molding, magnesium granulate, C-fiber, feeding problems, microstructure, intermetallic phase

1. INTRODUCTION

Thixomolding[®]

A special feature of magnesium injection molding (*Thixomolding*[®]) [1] assumes the processing of magnesium alloys in the solidus-liquidus solidification interval. In this temperature range, metals are suspensions showing liquid and solid phase constituents and a thixotropic¹⁾ behavior. Metals only behave thixotropically when the structure at the time of shaping is marked by the fact that solid particles with a higher melting point are embedded in an already liquid phase [2].

Previously manufactured cold magnesia granulates, or alloy chips are used as the starting material [3]. This is fed to a rotating screw at room temperature via a conveyor system and conveyed to the screw tip via a heating section. The screw is located in a cylinder with external heating. Since magnesium and its alloys easily oxidize in the liquid state, it is important that the cylinder is flushed with argon when heating up and when switching off or cooling down. Knowing that argon has a higher

density than air and can displace it, argon is particularly used in completely closed systems such as *Thixomolding*[®] machines.

Since the screw space is heated in a controlled manner over several temperature zones distributed over the length of the screw, the molding compound is heated particularly on the way to the screw tip through constant repositioning.

The dendrites of the solidified part of the molding compound are destroyed by constant shearing and a viscous paste with solid, rounded components is created. With a fast axial feed movement of the screw, the partially solidified melt is injected through the gate into a preheated metal mold at high speed (between 10 and 100 m/s) and high pressure (~ 80 MPa). At the beginning of a spraying process there is a cold plug in the nozzle, which serves as a closure and prevents the melt from running out in an uncontrolled manner or from oxidizing in the antechamber. The non-return valve has the task of blocking the screw antechamber to the screw during the press-in process. Only in this way can the full pressure build up in the anteroom and thus be transferred to the molten metal (specific casting pressure). After the mold has been filled, hold pressure is applied.

Draw-in problems with Thixomolding[®]

At *Thixomolding*[®], interference factors can occur that can lead to strong deviations from the

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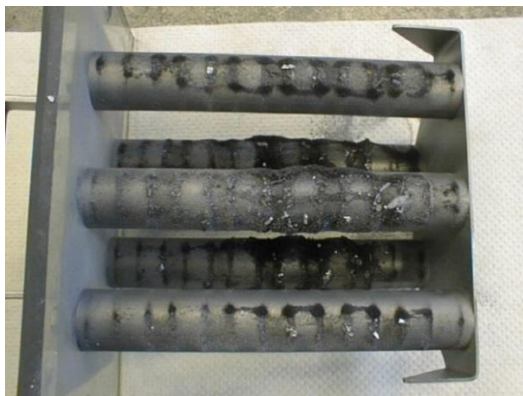
¹⁾ Thixotropy – time-dependent reduction of viscosity under stress

prescribed technological process parameters. The resulting process deviations can lead to operational disruptions in the intake or conveying area of the screw (so-called bridging). This is the most common fault that can occur in the initial phase when the alloy is changed and can lead to a longer downtime of the Thixomolding[®] machine.

According to Dworog [4], who investigated injection molding of magnesium alloys AZ91D and AM60, the following causes will lead to bridging:

- too high leaks at the non-return valve,
- defective non-return valve, e.g. sheared screw tip,
- rough surface of the screw,
- unsuitable screw or screw geometry,
- defective screw flanks,
- oxidation of the magnesium alloy due to defective argon supply, argon supply switched off too early or switched on too late, empty gas bottle or similar,
- defective heating bands,
- low heating power with high shot weights,
- Unsuitable geometry of the filling opening,
- unsuitable granules,
- unsuitable temperature profile,
- approach strategy unsuitable.

Czerwinski [5] reports on the formation of bridges by agglomeration of the magnesia granulate in the screw. The cause was named aluminum segregation, which can lead to a local increase in the melting temperature and, as a result, to uneven heating of the granulate.



a)



b)

Figure 1. Material contamination on the magnetic separator of the granulate bunker

Slika 1. Kontaminacija materijala na magnetnom separatoru bunkera za granulat

The unevenness of the granulate, unsuitable bulk density (over 0.72 g/cm³) and the dust content (particles <500 μm) can lead to the formation of

Experiments, results and discussions

When investigating magnesium injection molding in the solid-liquid state, we observed the influence of disruptive factors that could impair the course of the Thixomolding[®] process. Our investigations with magnesium alloys AZ91D, ZC63 and MRI230D have shown that in addition to the known causes of draw-in disorders, there are other important factors that could lead to bridging during Thixomolding[®]. These are:

- contaminated granulate
- unevenness and size of fine particles
- low melting eutectic
- chamfers or particles that are used to reinforce magnesium alloys.

The causes of disturbance factors that occur have been analyzed and solutions have been developed that can eliminate and avoid them.

Contaminated granules

An important prerequisite for the smooth implementation of the magnesium injection molding process is the use of pure magnesium granulate. Nowadays the manufacturers offer the granulates in various forms and qualities for the Thixomolding[®] process [3]. The granulate, which can contain metallic fragments as well as dust-like particles, can lead to considerable feeding problems. The dusty particles can lead to malfunctions, especially in the non-return valve. Figures 1 and 2 show ferromagnetic impurities that are separated from the magnetic separator as well as from cylindrical permanent magnets and all come from a barrel with the granulate of the alloy MRI230D.

bridges as well as to problems with feeding and conveying the screw. The size of the fine particles should be low (up to 5%), which is necessary for safety and processing reasons [6, 7].

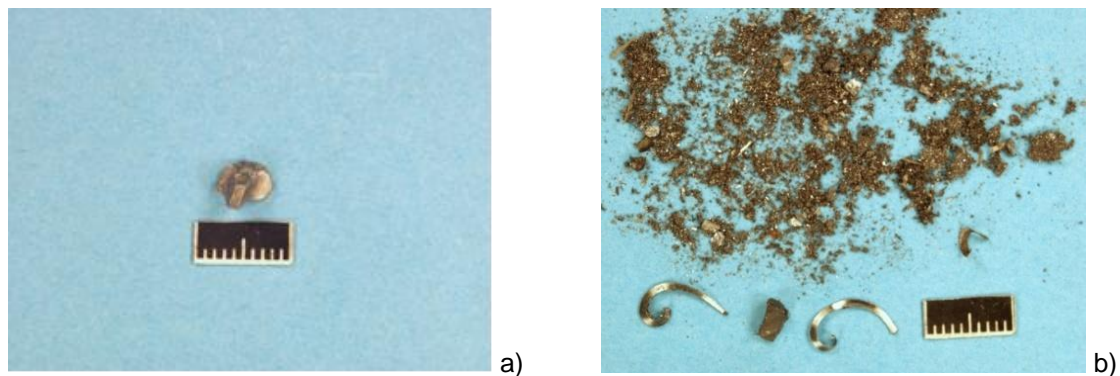


Figure 2. Ferromagnetic impurities in alloy MRI230D

Slika 2. Feromagnetne nečistoće u leguri MRI230D

Bridging by the carbon fibers

The properties of pure metals and technical alloys often reach their limits, which limits their further application. However, this can be improved by using different reinforcing fibers or particles [8].

To investigate the joint processing of magnesium alloys and carbon fibers (C fibers) in the Thixomolding® process, the investigations were

carried out with alloys AZ91D, ZC63 and MRI230D with short carbon fibers (C content > 95%) of the type SIGRAFIL C25 M350 UNS. SIGRAFIL short carbon fibers are characterized by increased flowability and good mixing and processing properties. Magnesium granulate (Fig. 3a) and carbon fibers (Fig. 3b) with a diameter of 8 μm and a length of 250 to 450 μm were used as starting materials for the composite production.

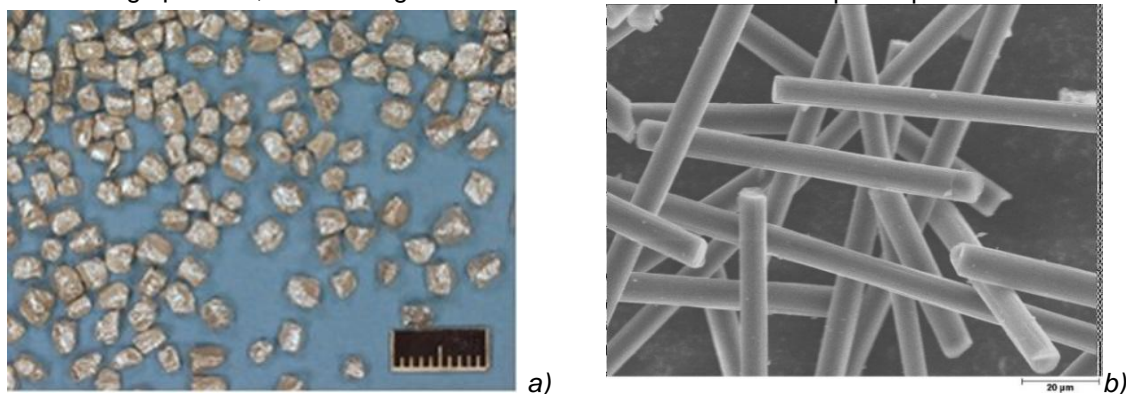


Figure 3. Starting materials for composite production: a - Magnesium granulate (size 2000-4000 μm); b - SIGRAFIL C25 M350 UNS carbon fibers (SEM image)

Slika 3. Početni materijali za proizvodnju kompozita: a - Magnezijum granulat (veličine 2000-4000 μm); b - SIGRAFIL C25 M350 UNS karbonska vlakna (SEM slika)

The fiber metering device proved to be capable of automatic, continuous fiber addition by means of a fiber metering device integrated in the Thixomolding® machine, so that the addition of metering aids (e.g. fumed silica) could be dispensed with. Since the density of the C-fibers almost corresponded to the density of the magnesium granules, a volumetric dosage was almost the same as a weight-proportional fiber admixture. The dosing system made it possible to mix a defined quantity of fibers into the magnesium granulate falling stream, which then together pass through the plasticizing unit into the screw antechamber and are conveyed further. When the metering device was put into operation, fiber

admixtures of up to 70% of the maximum delivery rate of the metering device were achieved, which corresponded to a fiber content of approx. 12-15% by weight in the magnesium alloy. The processing of the C-fibers in the parallel dosing operation between magnesium granulate and the addition of C-fibers takes place on the outside of the cylinder without any problems. Both dosing systems worked independently of each other and defined amounts of magnesium granulate or carbon fibers were introduced into the downflow of the cylinder opening.

The tests carried out showed that it is possible to jointly process magnesium granules and carbon fibers of the type SIGRAFIL C25 M350 UNS in the

Thixomolding® process. Using a suitable metering device, carbon fibers can be continuously mixed and processed in the granulate feed into the plasticizing unit. At the appropriate temperatures and injection pressures, there is a good fiber connection to the magnesium matrix in the component.

The investigations were carried out with different amounts of fiber between approx. 1 and 15% by weight, which resulted in a variation in the

amount of fiber due to the installed screw conveyor for carbon fibers.

Figure 4 shows a uniform orientation of the short fibers in the direction of flow of the magnesium mass from AZ91, whereby it is naturally only possible to distribute the short fibers in the liquid part of the magnesium mass. The fiber volume content is calculated using the relationship between the cross-sectional areas of the fiber.

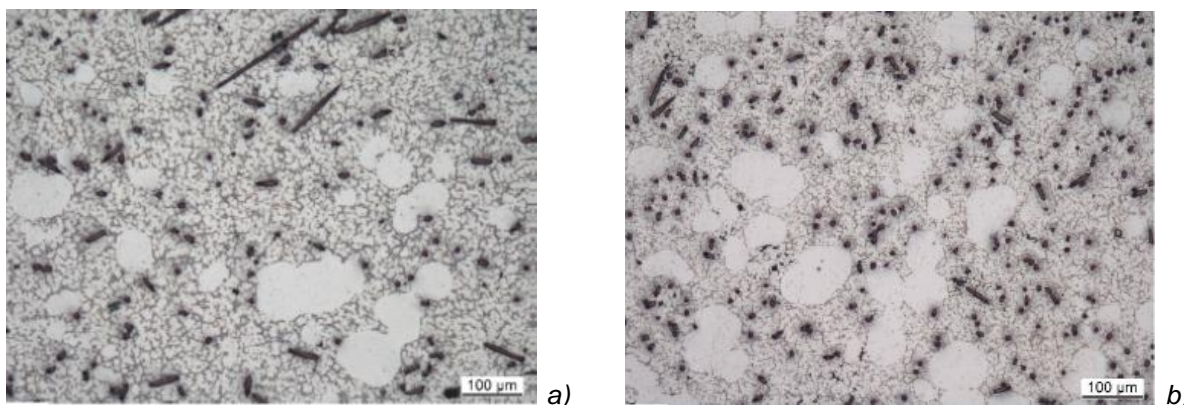


Figure 4. C-Fiber distribution in AZ91D (cross-section): a) 40% dosing rate of the fiber doser (proportion approx. 8% by weight), total area of the C fibers 6%; b) 60% dosing capacity of the fiber feeder (proportion approx. 12% by weight), total area of the C fibers 8%

Slika 4: Raspodela C-vlakana u AZ91D (poprečni presek): a) 40% kapaciteta doziranja dozatora vlakana (udeo oko 8% po težini), ukupna površina C-vlakana 6%; b) 60% kapaciteta doziranja dozatora vlakana (udeo oko 12% po težini), ukupna površina C-vlakana 8%

At the appropriate temperatures and injection pressures, there is a good fiber connection to the magnesium matrix in the component. However, the flow geometry of the non-return valve for fibers still

needs to be taken into account, as C-fibers cannot be mixed with the alloys in unlimited quantities and malfunctions in the non-return valve occur after a certain number of continuous spraying operations.

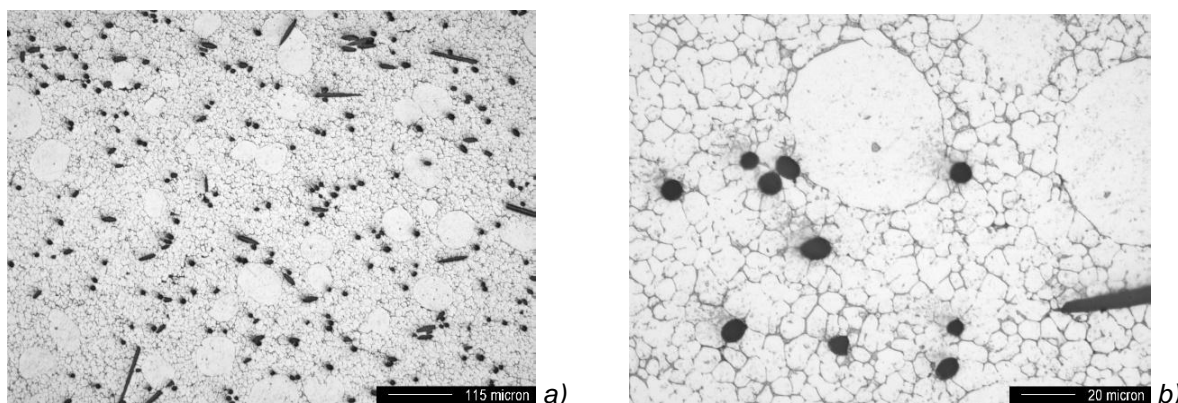


Figure 5. Fiber distribution in ZC63, 60% dosing capacity of the fiber feeder. Total area of the C-fibers 5.5% (cross-section)

Slika 5. Distribucija vlakana u ZC63, kapacitet doziranja dozatora 60%. Ukupna površina C-vlakana 5,5% (poprečni presek)

Figure 5 shows a homogeneous C-fiber distribution in the structure of a test specimen made from the alloy **ZC63** with a solid content of 9% and 11%.

During the processing tests with ZC63 including the fiber dosing, difficulties arose due to frequent bridging and blockages of the screw (Fig. 6).



Figure 6. Bridge in the retraction of the cylinder.
Pulling the screw at 735°C

Slika 6. Most pri retrakciji cilindra. Povlačenje šrafe na 735°C

As the processing time progressed, the C-fibers and the magnesium granulate were jammed in front of the non-return valve, as the C-fibers could no longer pass through the narrow opening cross-sections of the non-return valve. In order to remove the screw blockages or to be able to remove the screw for cleaning the plasticizing unit, the cylinder temperatures had to be increased up to 700°C. This led to critical conditions for the Stellite 6 wear protection layer on the inside of the cylinder and the outer contour of the screw, which contained approx. 60% cobalt.

Magnesium practically does not interact with high temperature melting transition metals such as chromium, molybdenum, tungsten, iron etc. Some high temperature melting transition metals - manganese, zirconium, nickel and cobalt - however, partially dissolve in liquid magnesium [9]. The liquidus temperatures in the Mg-Co system were determined by thermal analysis by Wetherill [10] and Cramer [11] and show good agreement. Magnesium and cobalt form a low-melting eutectic at 635°C. In the phase diagram shown, the eutectic point is 1.91 at.% Co (5 wt.% Co) (Fig. 7). The main component of the eutectic - intermetallic phase $MgCo_2$ (hexagonal C14-type binary Laves phase) - only dissolves at approx. 970°C [12].

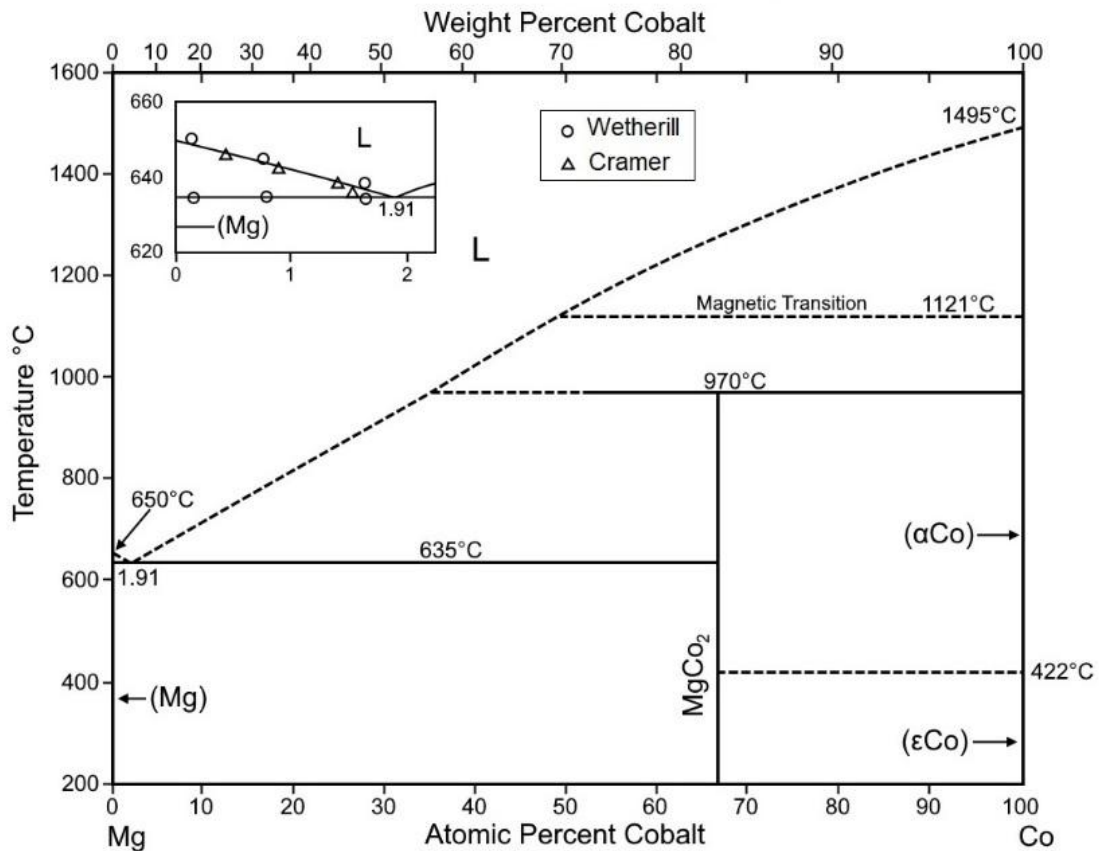


Figure 7. Mg-Co phase diagram [according to 13]

Slika 7. Fazni dijagram Mg-Co [prema 13]

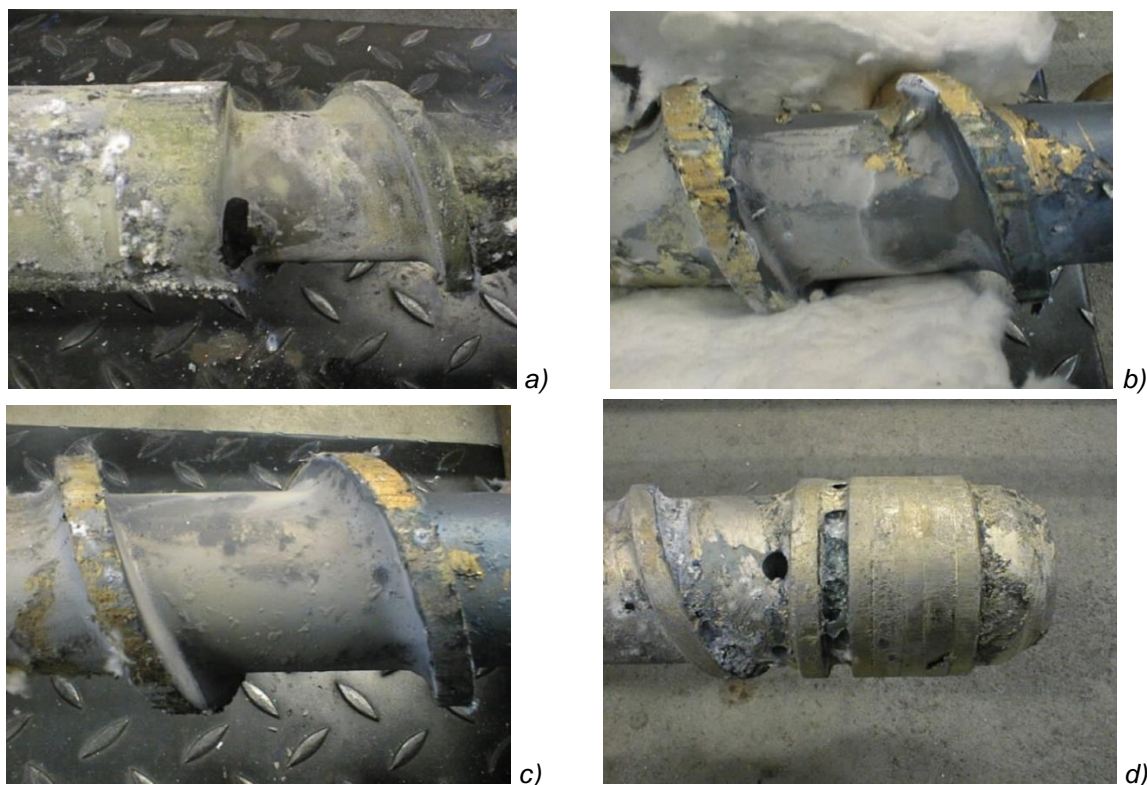


Figure 8. Screw after pulling at 735°C
Slika 8. Šrafe nakon povlačenja na 735°C

The condition of the screw, the non-return valve and the piston rings after use at temperatures above 700°C is shown in Figure 8.

The above conditions represent an increased attack of adhesion for the screw and the piston rings, which has led to a failure of the sealing effect of the piston rings. The high operating temperatures lead to a relaxation of the pretensioned piston rings and a sealing effect is no longer given. Thus, low-pore test bodies can no longer be produced.

The tests with C-fibers showed that the non-return valve used (screw tip) with the existing through-holes for C-fibers was problematic and was replaced by a fiber-appropriate non-return valve with larger openings.

The composite components made of **MRI230D/C** fiber were produced at 600°C and at a constant speed profile of 150/180/180/150 cm/s. The C-fibers were evenly distributed over the entire cross-section of the sample (Fig. 9).

The manufacturing parameters used resulted in very good and complete adhesion between C-fibers and matrix. The C fiber volume fraction within the samples (cross-section) was approx. 8% in the middle and approx. 7% at the edge (Table 1).

After the 28th injection process, bridging occurred. By means of online data acquisition on

the Thixomolding[®] machine, any process parameter deviations that occurred during the bridging process were recorded. The following images show an example of the process parameter deviations from a stable process (Fig. 10) that were registered during the bridge formation (Fig. 11) and after the bridge formation (Fig. 12) in the tests with MRI230D and carbon fibers.

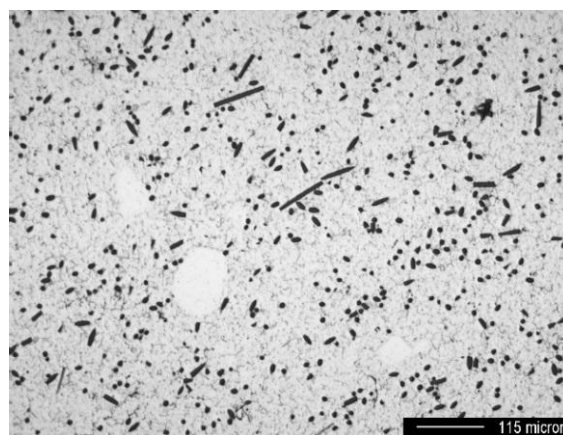


Figure 9. Distribution of the solid content and C-fibers over the cross-section of a composite sample made of MRI230D + SIGRAFIL C25 M350 UNS

Slika 9. Raspodela čvrste faze i C-vlakana po poprečnom preseku kompozitnog uzorka MRI230D + SIGRAFIL C25 M350 UNS

Table 1. C-fiber distribution statistical data

Tabela 1. Statistički podaci o raspodeli C-vlakana

| Measurement range | Fiber content (Vol.-%) | Number of fibers (per mm ²) | Number of evaluated fibers | Fiber surface (μm ²) | Fiber length (μm) | Fiber width (μm) | Elongation |
|-------------------|------------------------|---|----------------------------|----------------------------------|-------------------|------------------|------------|
| Edge | 7 | 632 | 1865 | 108,3 | 12,8 | 7,9 | 0,6 |
| Center | 8 | 750 | 2215 | 111,3 | 13,1 | 8,0 | 0,7 |

The graphical representation of measured values of immediately consecutive injection processes shows the occurrence of a fault. In the course of this sequence, a bridge is formed (Fig.

12), in which both the height and the characteristics of the metal print (light blue) change. The peak values per injection process remain well below the values before the bridge formation (see Fig. 10).

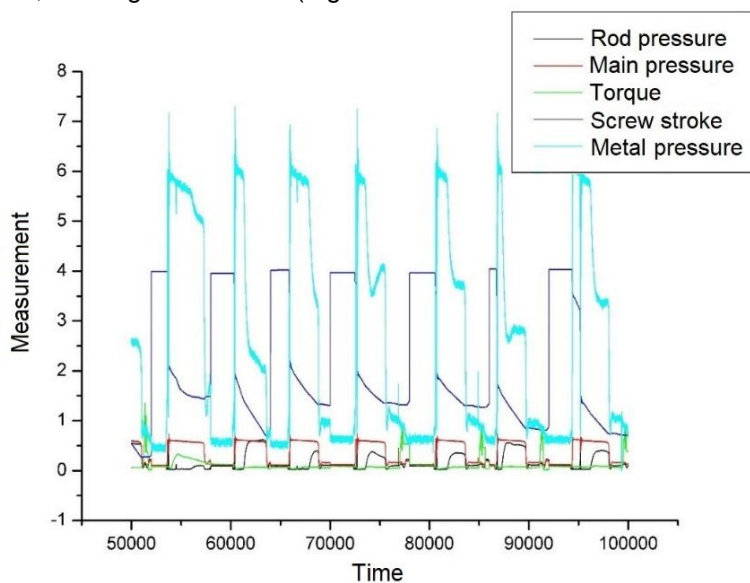


Figure 10. Experiments with MRI230D and carbon fibers. Process parameters before bridging
Slika 10. Eksperimenti sa MRI230D i karbonskim vlaknima. Parametri procesa pre premošćavanja

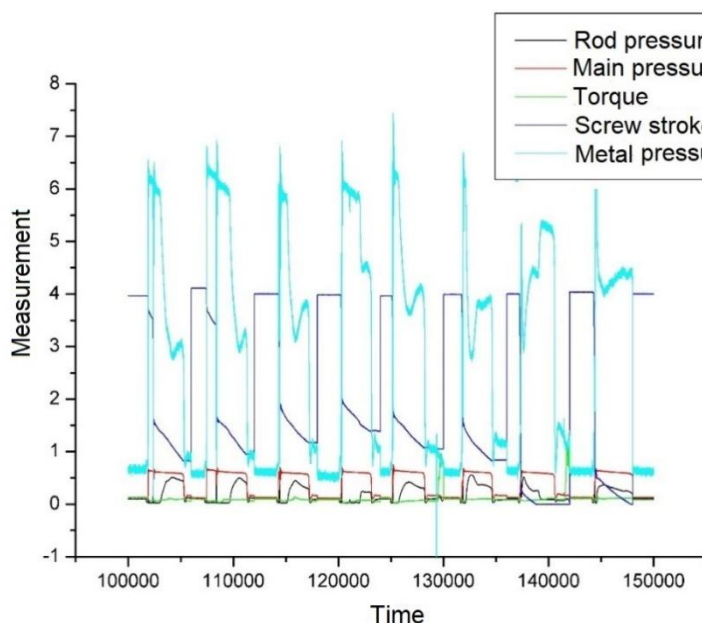


Figure 11. Experiments with MRI230D and carbon fibers. Process parameters for bridging
Slika 11. Eksperimenti sa MRI230D i karbonskim vlaknima. Parametri procesa za premošćavanje

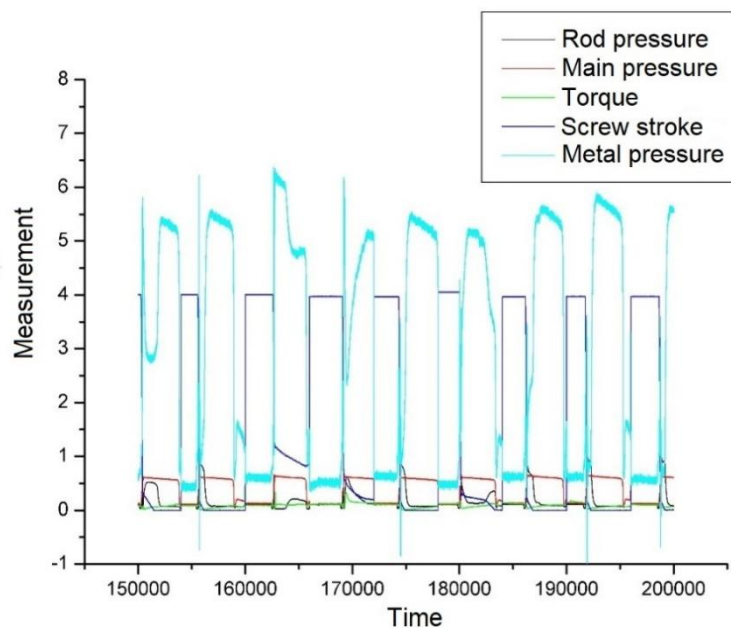


Figure 12. Experiments with MRI230D and carbon fibers. Process parameters after bridging
 Slika 12. Eksperimenti sa MRI230D i karbonskim vlaknima. Parametri procesa nakon premošćavanja

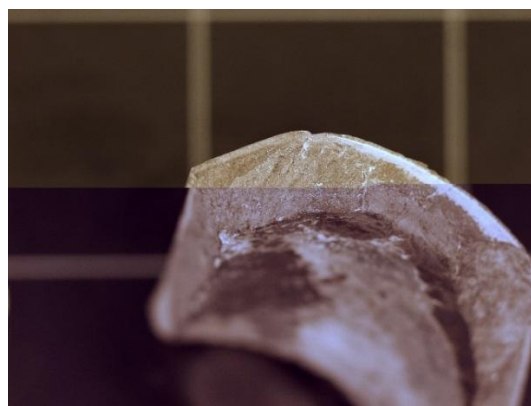
The characteristic curve progression during the injection process (sharp rise, short stop, slight drop, stop, drop to the original level) can no longer be determined after the bridge has been formed (Fig. 12).

It was no longer possible to restore the process parameters, which enable a further normal

production process, until the screw was removed from the machine and cleaned or replaced. After the screw had been dismantled, the samples were taken from the barrel and the web area of the screw (Fig. 13) and examined using a light and scanning electron microscope.



a)



b)

Figure 13. Material from the screw flights. The marking shows the point at which the specimens were taken (a) and the fracture surface in the transverse direction (b)

Slika 13. Materijal sa šrafova. Oznaka pokazuje tačku na kojoj su uzorci uzeti (a) i površinu loma u poprečnom pravcu (b)

The C-fibers are distributed inhomogeneously. The edge layers of the sample pieces (Fig. 14a) have a lower C-fiber content (up to 10%) than in the middle of the sample (Fig. 14b) - approx. 50%

C-fibers. The sharp separation between the two areas can be clearly seen in image 15c. Figure 15d shows the agglomerated carbon fibers that led to the formation of bridges.

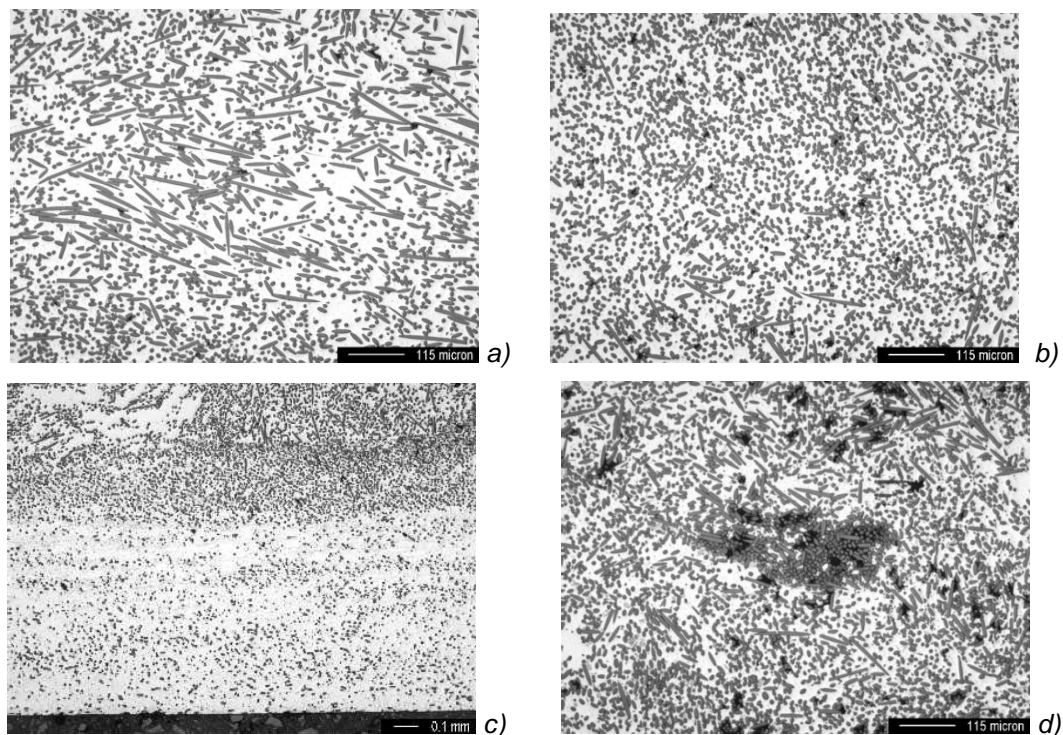


Figure 14. Distribution of the C-fibers at the edge of the examined sample piece (a), fiber agglomerate (b). Longitudinal grinding

Slika 14. Raspodela C-vlakana na ivici ispitivanog uzorka (a), aglomerata vlakana (b). Uzdužno brušenje

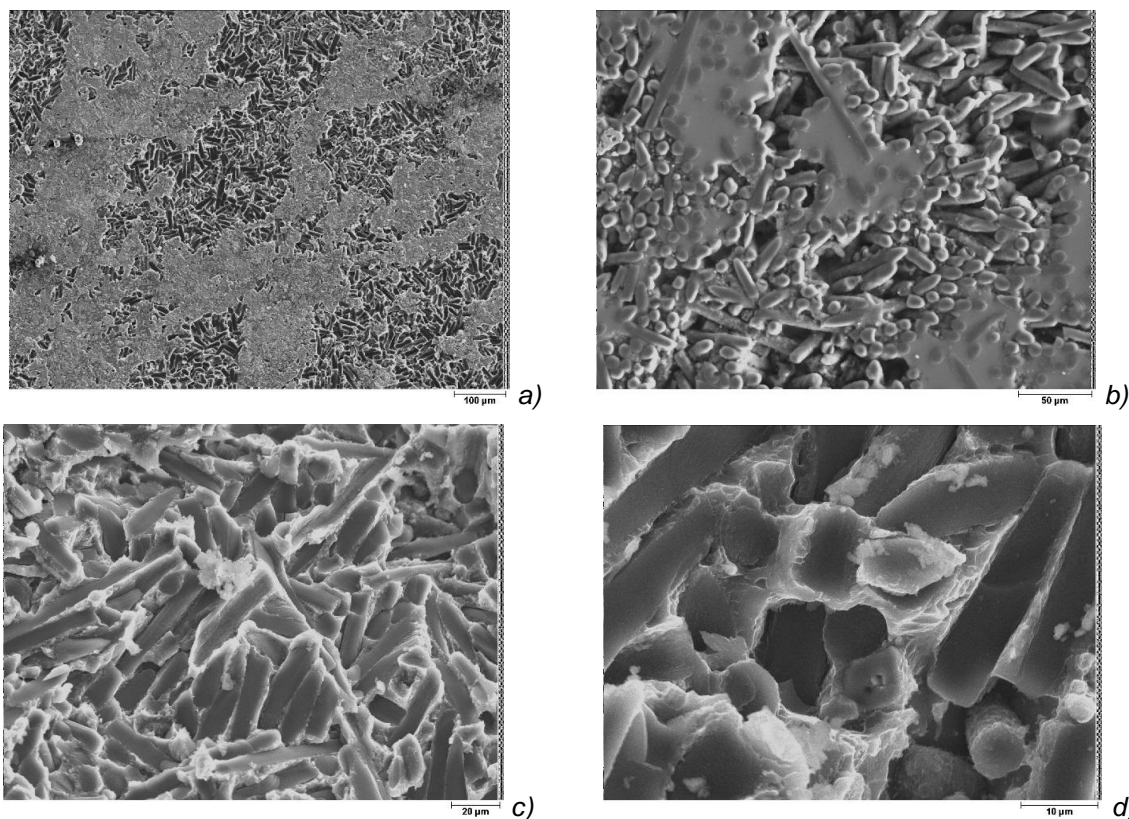


Figure 15. MRI230D with C-fibers: fracture surface of the cylinder contents after bridging (SEM images)

Slika 15. MRI230D sa C-vlaknima: površina loma sadržaja cilindra nakon premošćavanja (SEM slike)

In all samples, however, there was more agglomeration of the C fibers in the middle than in the edge area (Table 2).

Table 2. Cylinder content after bridging

Tabela 2. Sadržaj cilindra nakon premoščavanja

| Characteristic | Longitudinal grinding | | Cross-section | |
|-----------------------------------|-----------------------|--------|---------------|--------|
| | Edge | Center | Edge | Center |
| Surface (μm^2) | 76,8 | 310,8 | 439,8 | 439,8 |
| Elongation | 0,7 | 0,7 | 0,7 | 0,7 |
| Fiber length (μm) | 9,9 | 40 | 56,3 | 56,3 |
| Fiber thickness (μm) | 6,9 | 7,3 | 7 | 7 |

Figure 15 shows the fracture surface of the cylinder contents after bridging. A non-uniform sheathing of C-fibers by the MRI230D magnesium alloy can be clearly seen.

CONCLUSION

It has been established that in addition to the known causes of the draw-in disturbances that can occur in the initial phase when the alloy is changed, other important factors such as the use of contaminated magnesium granulate, uneven size of fine particle proportions, formation a low-melting eutectic and the fibers and particles used to reinforce magnesium alloys lead to the formation of bridges.

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IZVOD

POREMEĆAJI PRI INJEKCIJONOM BRIZGANJU LEGURA MAGNEZIJUMA

Istraženi su i objašnjeni problemi pri injekcionom brizganju različitih legura magnezijuma. Ispitivanja sprovedena sa legurama magnezijuma AZ91D, ZC63 i MRI230D su pokazala da pored poznatih uzroka, na poremećaj pri injekcionom brizganju mogu uticati i drugi faktori, kao što su kontaminacija granulata, neravnine i veličina finih čestica, eutektikumi sa niskom tačkom topljenja i vlakna koja se koriste za ojačavanje legura magnezijuma. Analizirani su uzroci faktora poremećaja koji nastaju i razvijena su rešenja za njihovo otklanjanje i izbegavanje.

Ključne reči: brizganje, magnezijum granulat, C-vlakno, doziranje, mikrostruktura, intermetalna faza

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