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Processability of Mg-Gd powder via friction extrusion

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Abstract. Friction extrusion (FE) is a solid-state processing technique based on heat and shear introduction via friction at the die-feedstock interface. FE can be used to process feedstock of different forms, such as solid billets, chips or powder as well as being applicable to a variety of Al-, Mg- and Cu-alloys. The relative rotation between die and feedstock makes preheating obsolete and the induced plastic deformation has the potential of not only energy-efficient consolidation but also grain refinement for improved extrudate properties.

In this study, the processability of Mg-Gd powder via FE is investigated. Mg-Gd alloys have their main use in aerospace as well as being promising candidates for biomedical applications. In these fields the homogeneity in terms of mechanical and chemical properties, respectively, is a critical factor.

Consequently, the application of FE in this study aims to provide insight on a new possible processing route for Mg-Gd alloys by investigating the mechanical properties of the extruded, fully consolidated wires, i.e. hardness and compressive properties, as well as the microstructural features of the feedstock during processing, i.e. void volume, grain structure and morphology changes.

Keywords: Friction extrusion, Magnesium alloy, Powder consolidation.

1 Introduction

Friction extrusion, first patented by Thomas et al. [1] in 1993, describes the application of axial force and relative rotation by a die onto feedstock to generate an extrudate. The technology, based on friction-induced heat generation and high imposed shear deformation, allows for fully consolidated and recrystallized extrudates of various metals by plasticization and forming without the need for pre-heating. An essential factor for controlling the properties and in particular the homogeneity of grain refinement in the extrudate constitutes the mechanism of shear introduction from the die into the feedstock [2].

The successful friction based processing of Mg-alloys from bulk feedstock into tubes has been reported for AZ31 [3] and ZK60 [4] and found to be an energy-efficient processing route [5]. Another investigated application is the recycling of Mg-chips feedstock into wires from pure Mg [6] and AZ31 [7] with effective consolidation reported. The analytical focus on homogeneity, e.g. in AZ91E tubing [8], or corrosive properties [9], shows promising properties of the extrudates, although process limitations were reported for ZE41 wire extrusions [10], AZ31 [11] and pure Mg [12]. The limitations were found to be mainly related to process control dependent temperatures and strain rates achieved locally during processing.

Next to FE, similar solid state extrusion processes like CoreFlow [13] or KOBExtrusion with the forward-backward rotating die method [14] have been used to process Mg, confirming the main challenges, which are achieving a controlled microstructure and homogeneity in the extrudate as well as ensuring the stability of thermal and time-dependent conditions during processing. Overall, these investigations are highlighting the interest in the studied processing technique FE.

By using powder feedstock, the high potential to achieve superior ductility or tailored microstructural features was shown by Whalen et al. [15] in FE from aluminum alloy and a wide range customization in conventional extrusion was demonstrated for Mg-Gd alloys by Harmuth et al. [16]. Hence, this study aims to give a first insight into the application of FE to process Mg-Gd powder feedstock with the perspective of opening a new processing route for tailored semi-finished products for aerospace or biomedical applications.

2 Experimental Methods

The friction extrusion setup and process parameters as well as the analytical methods applied to evaluate extrudate quality are described. Special attention is given to the feedstock preparation due to the influence on process-critical initial friction and consolidation conditions.

2.1 Feedstock

For each extrusion 100 g of MgGd5 powder with spherical particles between 36 and 150 μm were filled into the container and pre-compressed on a hydraulic press at 50 kN. The MgGd5 powder consists of a mixture of an Mg-Gd alloy with 6.8 wt.% Gd and pure Mg particles. To prevent powder spillage at the beginning of the process and to ensure controlled initial friction and pressure conditions, the compressed feedstock was capped with 1 mm foil of pure Mg as well AZ31. The intention hereby was to enforce even consolidation and heating in the process until the onset of plasticization. After installing the container in the FE machine, the capped powder is further compressed without rotation up to 100 kN, resulting in 40 mm depth of feedstock in the container.

2.2 Friction extrusion

The processing was performed on the friction extrusion machine FE100, Bond Technologies, utilizing an AISI 4140 container with 50 mm inner diameter and featureless, flat faced AISI H13 dies with 10 mm die bore, i.e. an extrusion ratio of 25, and 8 mm land length. The wires were extruded into a closed guiding tube and flushed with Argon to minimize oxidation. After initial parameter studies, 10 processes were performed under force control at identical control parameter settings. 4 extrusions to 30 mm feedstock depth used pure Mg cap material, 6 processes with 10 to 30 mm depth in 5 mm increments used AZ31 cap material. At the initial processing stage, a pre-heating phase with 55 kN axial force at 300 rpm was used to facilitate the beginning of extrusion, which was initiated after on average 7 s of contact during 2.8 mm of die advance. Hereby the axial force was increased to 100 kN over a 10 s ramp time and the rotation kept constant at 300 rpm. The temperature was measured via a thermocouple placed 1.5 mm behind the die face at a radius of 16.5 mm. After reaching the targeted plunge depth into the feedstock material, the die was retracted under rotation.

2.3 Analysis

Analysis of the machine feedback is focusing on die temperature and plunge rate as main response parameters. The extruded wires and residual feedstock material were cut, cold mounted, ground and polished. Microscopic analysis was performed using a VHX-6000, Keyence, light optical microscope. For investigation of microstructural changes on higher resolution, a Quanta 650 scanning electron microscope (SEM), FEI Company, equipped with concentric backscattered (CBS) detector was used. In order to track the introduction of cap material into the wire and detect larger volumetric defects, X-ray micro-computed tomography (microCT), an Y.Cougar SMT, YXLON International, was employed.

For mechanical characterization of the extrudates micro-hardness mapping according to ASTM E384-22 with a load of 0.2 kgf is performed on a DuraScan G5, Struers. Compression testing following ASTM E9-19 is used to measure the mechanical properties of full wire sections (F) as well as machined wire core specimens (C) with a length/diameter ratio of 1.5 at a traverse speed of 0.012 mm/s. The specimens with 14.5 mm (F) and 12 mm (C) length, respectively, are tested on a universal testing machine, ZwickRoell, with a 100 kN load cell.

3 Results and Discussion

The processability of Mg-Gd powder via FE is evaluated on different levels, addressing aspects relevant for production and further use of the extrudates. In terms of processing behavior, the process stability, temperature evolution as well as continuity of material flow are considered. Due to the need of eliminating the void volume in the feedstock, the powder morphology changes and the achieved microstructure in the extrudates are discussed in detail. Furthermore, the mechanical properties investigated

by indentation and compression tests are evaluated with respect to the achieved homogeneity in the extrudates.

3.1 Processing Behavior

Extruded wires. All processes resulted in full-length solid wires of up to 550 mm length. The wire diameter ranges from 9.8 mm in the beginning and down to 9.6 mm towards the end of the extrusion. An example of extruded wire is shown in Fig. 1a. As seen in Fig. 1b, no sticking of the extruded material to the land length occurs as opposed to the outside of the die, where feedstock is pressed towards the container wall. The surface of the wire shows typical tooling marks correlating with the applied differential rotation. In the end sections most wires show serrated surface cracks of maximum 40 μm depth along the tooling marks, as shown in Fig. 1c.

Effect of cap material. Both cap materials used to facilitate the onset of extrusion were found to be effective. Typical results of insufficient pressure, e.g. chevron burst defects, as a consequence of low compacted feedstock or discontinuous material flow were avoided. Fig. 1d shows a microCT image of a wire tip with a typical distribution of cap material along the wire, visible as brighter area in the first 9 mm as well as a thin cladding on the wire circumference, gradually reducing in thickness during extrusion. Furthermore here the elimination of volumetric defects after less than 10 mm of extrusion is clearly seen. A better consolidation was achieved early on with AZ31 cap material due to its higher strength

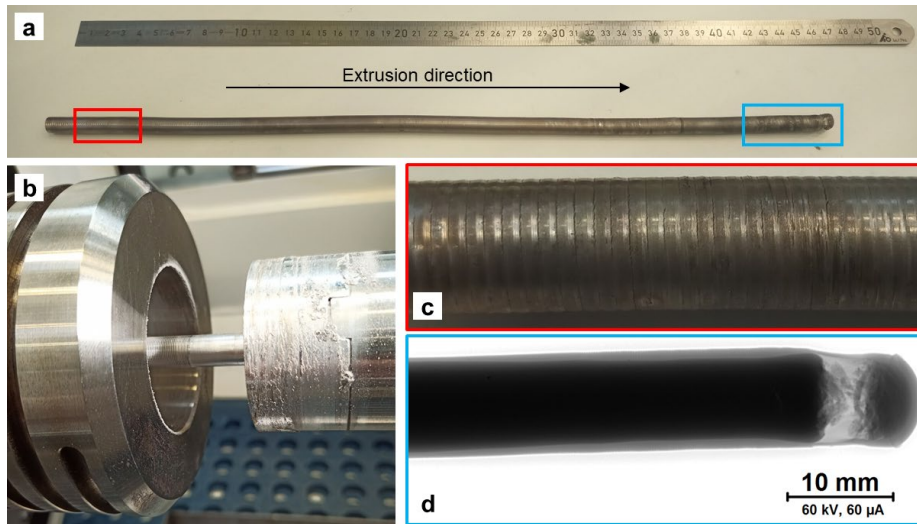


Fig. 1. a) Full-length wire with typical appearance, b) Wire in extrusion setup after die retraction, c) Surface cracks towards end of the wire and d) MicroCT image visualizing defects and distribution of cap material (AZ31, brighter area) at the wire tip and circumference.

One key factor to a successful extrusion in the beginning of the process is the initiation of friction at the die-cap interface. In two individual processes, one of each cap material, this could not be achieved and the cap stuck to the die, causing friction and plasticization at the cap-feedstock interface. This led to cap material being distributed along the whole length of the extrudates as opposed to being limited to the first 50-80 mm as shown in Fig. 1d. These two processes showed a significantly reduced extrusion rate and have been excluded from further analysis.

Process Stability. The processes showed good reproducibility in transitioning from pre-heating to extrusion phase, reaching early on steady conditions. A major influencing factor on both die temperature, Fig. 2a, as well as the corresponding extrusion rate, is found to be the cap material. Fig. 2b shows the die advance with approximately 2.8 mm of compaction during pre-heating stage and consequent 5-7 mm of further feedstock consolidation upon applying extrusion parameters.

The higher initial die advance rates are attributed to powder compaction, which was found to be higher for pure Mg cap material, despite lower recorded temperatures. This indicates a more efficient interlocking of the cap material with the powder feedstock as a result of the lower material strength of pure Mg compared to AZ31. Furthermore, a measurable increase in die advance after around 80 s processing time is observed for processes with AZ31 cap material. This can be a result of the initially lower heat dissipation by material flow, i.e. extrusion rate, and the effect of reduced feedstock volume towards process end, further affecting temperature gradients in front of the die. All processes stabilized in die temperature just above 600 °C, reaching a calculated extrusion rate of 6-12 mm/s, disregarding any still ongoing feedstock consolidation at this stage.

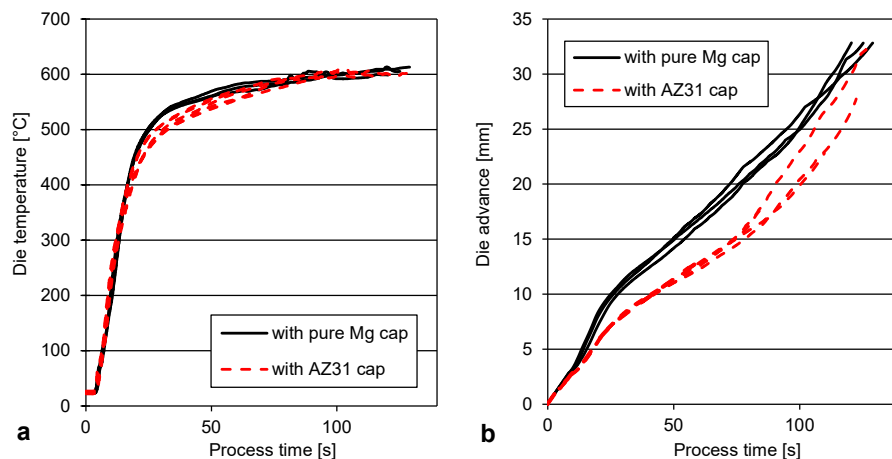


Fig. 2. a) Evolution of temperature at the die face and b) die advance over processing time for extrusions from Mg-Gd powder feedstock with pure Mg (black) and AZ31 (red) cap material.

3.2 Powder Consolidation

Feedstock Compaction. The powder in front of the die shows three distinct, convex shaped zones, characterized by different porosity densities. In Fig. 3a, the transitions between these zones are marked by red lines. Up to a maximum distance of 15 mm from the die face, the powder is virtually free of any void volume; individual particles are deformed but can be clearly identified. The shape of these particles is elongating at the inflow region and gradually broken up in the immediate proximity of the die face, with this effect being observed up to a depth of 4 mm at a radius of 6-10 mm. Further from the die face, up to a depth of 23 mm void volume is still present, but particles are compacted by deformation from their originally spherical shape. Deeper into the feedstock material, this spherical shape is intact and significantly higher void volume present. These zones establish during the first 10 mm of die plunge and remain stable in shape and extent throughout the process until effects of interaction with the container backing come into play.

Morphology Changes. Via SEM the changes in microstructure in terms of particle deformation as well as precipitate distribution are tracked. In Fig. 3b-e the evolution of powder particles passing the thermo-mechanically affected zone in front of the die is presented with powder compaction, particle elongation and final breaking up of the particle structure.

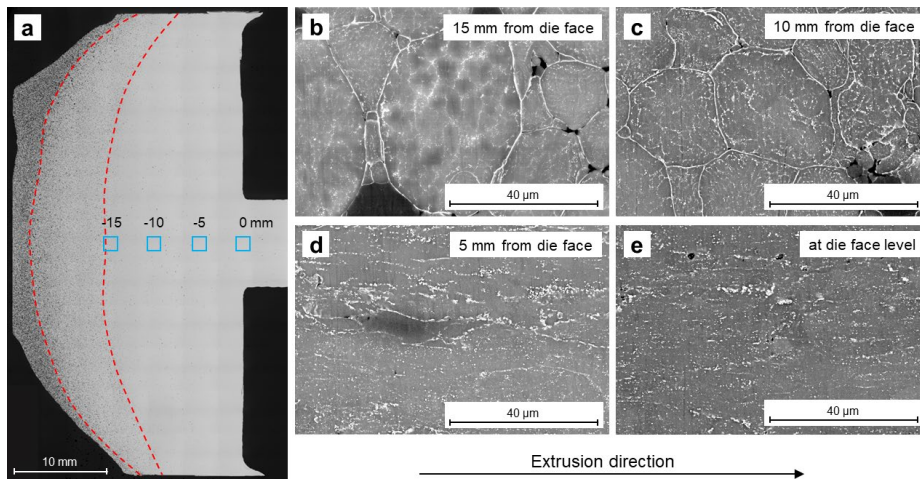


Fig. 3. a) Macrograph of wire and residual feedstock with indication of changes in void volume (red) and positions for SEM analysis (blue). Backscattered electron images of the feedstock at a distance of b) 15 mm, c) 10 mm, d) 5 mm and e) 0 mm from the die face.

In Fig. 4, the comparison between compacted feedstock (a) and extruded wire (b, c) is shown. No cap material is present in the wire anymore at this processing stage. Here clearly the initially near spherical pure Mg and Mg-Gd particles are shown with precipitation (bright phase) distribution concentrated along grain boundaries and particle

surfaces. In the wire center, Fig. 4b, the particles have undergone severe elongation. The deformation in extrusion direction (left to right) is dominant; individual particle surfaces can no longer be identified, although the powder components, Mg-Gd and pure Mg, are still distinguishable. In contrast to this, a single, homogeneous material structure is present in the outer parts of the wire, Fig. 4c. Homogeneous secondary phase distribution at higher magnification indicates a promising result of material alloying via FE, induced by severe plastic deformation and high temperature exposure. Opposed to the core structure, the material flow, homogenization and refinement is dictated by rotation related deformation in radial direction.

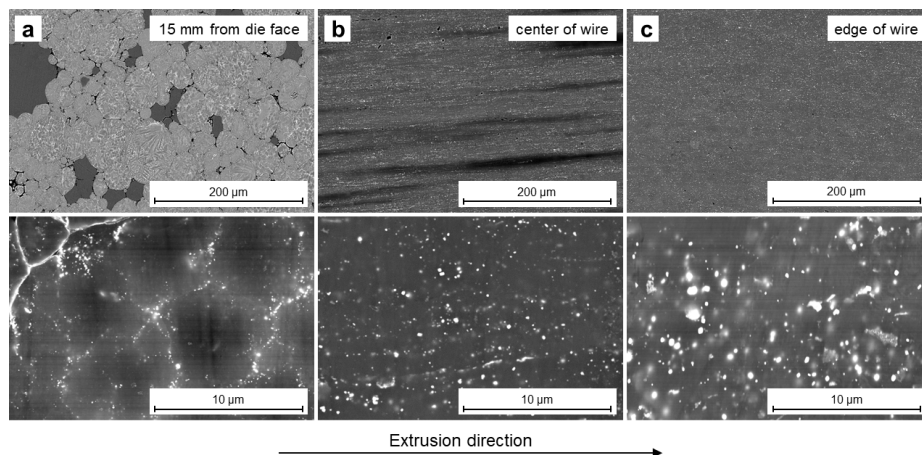


Fig. 4. Backscattered electron images obtained a) 15 mm in front of the die b) at the center and c) at the edge of the extruded wire. Positions marked in Fig. 3a.

Extrudate Density. On wire specimens taken from different processes as well as along an individual wire, no significant differences in material density were found. The exceptions here are the first sections of the wires, with remaining cap material and in consequence lower density. In average a density of $1.835 \pm 0.004 \text{ g/cm}^3$ was determined for the wire material. In comparison, a conventionally extruded reference wire MgGd5 showed a density of 1.808 g/cm^3 in the same measurement setup, which is lower than the minimum measured in the friction extruded wires of 1.827 g/cm^3 . The overall higher density might be attributed to differences in Gd content, however, the consistency between processes and even more between start and end of the wires, accounting for the described changes in processing conditions, indicates a full consolidation as supported by the absence of visible voids.

3.3 Mechanical Properties

Micro-hardness. Vickers hardness mapping was performed on wire cross-sections at 60 and 470 mm distance from the wire tip. No systematic trend in hardness over the radius was observed, i.e. center to circumference of the wires. The results for both

cross-sections are given in Table 1 and show an increase in hardness over wire length of around 10 %.

Table 1. Micro-hardness in cross-sections of friction extruded wire from MgGd5 powder.

Position in wire	Hardness AVG	MIN	MAX	SD
60 mm	72.1 HV0.2	63.5	79.6	3.9
470 mm	79.8 HV0.2	70.7	88.4	3.9

Compression Testing. Two sets of specimens were tested under compressive loading. One set consists of machined specimens from the wire core (8 mm diameter, 12 mm length), eliminating influence of the rough, as-extruded surface. Since this removes the zone of the extrudate, which is affected by processing conditions the most, a second set of full wire sections (9.7 mm diameter, 14.5 mm length) has been tested.

When interpreting the stress-strain curves, Fig. 5a, it needs to not only be considered that the surface roughness required by standard is exceeded significantly, but also that the measured wire diameter is slightly overestimating the initially loaded cross-section due to grooves on the surface. All curves show good concordance with the average ultimate compressive strength being higher (455 vs. 442 MPa) and the yield strength being lower (195 vs. 202 MPa) for the full wire specimens. The compression to fracture ranges from 15.2 to 18.1 %. All specimens failed by brittle fracture after uniform plastic deformation of at least 12 %. The fracture angles are similar for all specimen at around 45° and the fracture surface presented no clear indication of a fracture origin, as shown in Fig. 5b and c.

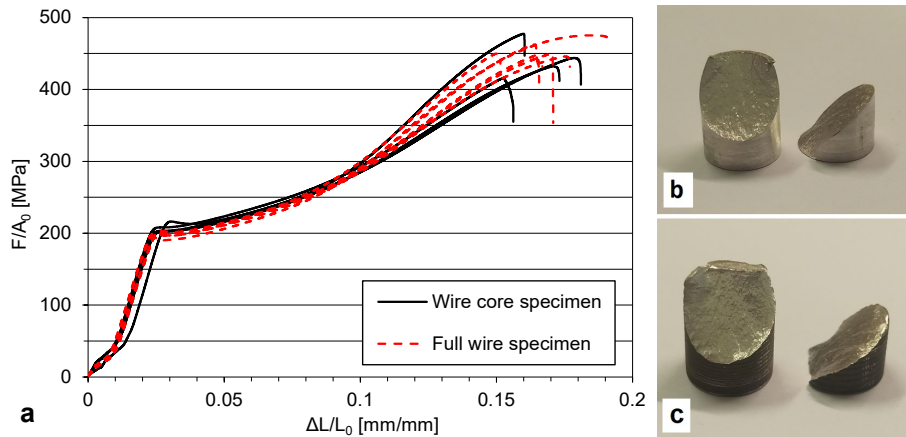


Fig. 5. a) Stress-strain diagram of the compression tests performed on the extruded Mg-Gd wires and the fracture surfaces of b) wire core and c) full wire specimens.

Compared to conventionally extruded MgGd5-wires [16], the malleability is on a rather low level, while the yield strength is in a comparable range. However, the ultimate strength has increased by up to 15 % compared to conventional extrusion. A

correlation of the measured values with the position in the extrudates is given in Fig. 6, showing highly constant properties along the wire, except for a slight decrease in ultimate compressive strength.

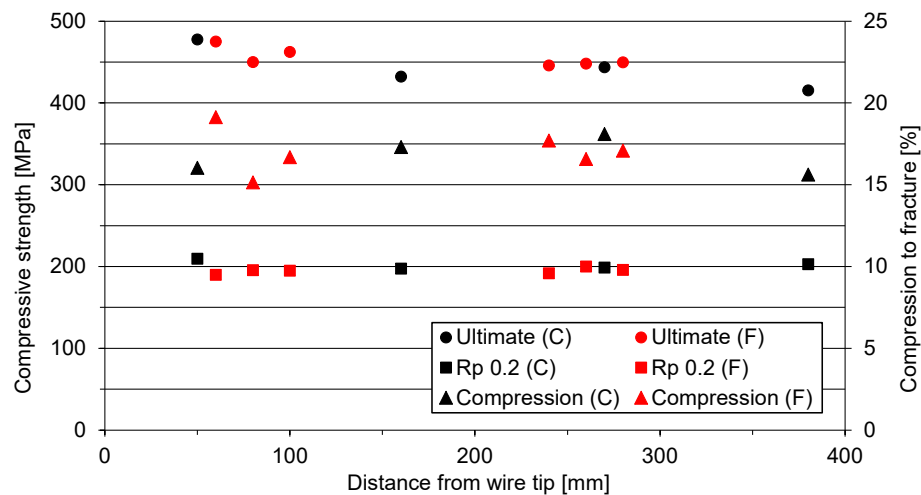


Fig. 6. Mechanical properties determined in compression testing for specimens from the wire core (black) and full wire section (red), over position in the extruded wire.

4 Conclusions

The presented study successfully utilized Mg-Gd powder to produce wires of 10 mm diameter via FE. With regard to possible applications, focusing on process stability, homogeneity and mechanical properties, the main findings conclude as follows:

- Fully consolidated wires of MgGd5 were produced from powder in single-step friction extrusion with a good reproducibility.
- In-process alloying from dissimilar powder feedstock and homogenization of secondary phase distribution was observed in parts of the wire.
- Homogeneous mechanical properties along the wire length were obtained, showing higher ultimate compressive strength, but lower malleability compared to results for conventional extrusion.
- Friction at the die-feedstock interface was identified as a critical factor for process control.
- The feasibility and effectiveness of introducing cap material for powder processing was investigated.

While the microstructural inhomogeneity remains in need of further optimization, the mechanical properties and achieved density are not affected hereby. Together with the achieved in-process alloying, the potential for producing wires with graded or tailored properties becomes visible, as well as offering an influencing factor for controlling

corrosion or degradation, as shown for WE43 [17]. Since a strong dependency on extrusion rate and processing temperature is known for Mg-Gd alloys [16], reduction in process temperature and an increase in strain can further refine the microstructure or alter mechanical or chemical properties. As especially these factors have been shown to cause defects in FE from AZ31 [11], a detailed look into texture development in the produced samples poses a consequent continuation of the presented work.

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Data Availability. The data related to this research is available online (<https://doi.org/10.5281/zenodo.7689070>).

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