

# Microstructural and mechanical properties of FAST/ SPS-compacted TRIP-steel-Mg-PSZ composites affected by the ball milling energy

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### Highlights

- The impact of the rotation speed during ball milling is highlighted.
- A Reduction of the grain size of the steel matrix is achieved by high-energy ball milling.
- An improvement of the compressive strength is obtained by high-energy ball milling.

## 1. Introduction

A composite material provides the opportunity to combine the positive properties of two or more different materials. Thus, the composite material exhibits superior properties compared to those of the single components. One example, which is in the focus of research, represents a composite consisting of a TRIP-steel matrix (TRansformation Induced Plasticity) reinforced with Mg-PSZ (partially stabilized zirconia) [1-4]. TRIP-steels exhibit a high strength as well as a high ductility due to a deformation-induced phase transformation from metastable austenite to  $\alpha$ '-martensite [5,6]. Furthermore, the Mg-PSZ is able to transform stress-induced from the tetragonal phase into the monoclinic phase. Thus, a volume expansion occurs, which is responsible for an enhanced strength within the composite material [7].

Spark Plasma Sintering provides the opportunity to achieve superior material properties. Due to the high heating rates, it is possible to compact materials by powder metallurgical route within low processing times. Thus, undesired grain growth is inhibited, which improves the mechanical properties of the material [8,9].

It is the aim of the present study to investigate the influence of varying rotation speed within the powder mixing process on the microstructure and compressive strength of the subsequent spark plasma sintered TRIP-matrix composites reinforced with Mg-PSZ.

# 2. Methods

The initial powders for the milling process are an austenitic TRIP-steel powder and a partially stabilized zirconia powder (Mg-PSZ, reinforced with 3.5 wt.% MgO). To receive the mixed powders (ceramic content of 5 vol.-% or 40 vol.%) for sintering, the initial powders are mixed for 4 hours with a ball to powder mass ratio (BPR) of 5:1 at a rotation speed of 100 rpm and 250 rpm in a planetary ball mill, respectively. After mixing, the composite powders were filled in graphite dies for SPS. A constant heating rate of 100 K/min and a linearly increasing pressure (up to 51 MPa) were applied until the maximal sintering temperature of 1100 °C (dwell time: 5 minutes) via SPS. Density measurements of the sintered composites by Archimedes method supported the calculation of the densification of the samples during sintering. The compressive tests were carried out with a strain rate of  $10^{-3}$  s<sup>-1</sup> using 6 mm x 6 mm (height x diameter) samples. Optical microscopy, scanning electron microscopy (SEM), X-ray diffraction and electron backscatter diffraction were applied for the microstructural investigation of the as sintered material and the compressed samples.

## 3. Results and discussion

The low rotation speed of 100 rpm results in an apparently homogeneous distribution of the initial powder particles without an observable deformation of the particles. However, the Mg-PSZ forms clusters. Applying high-energy mixing at 250 rpm leads to intensively deformed steel particles combined with a clamping of the ceramic to the steel particles. This effect is reflected in the microstructure of the bulk composite material. SE-micrographs of samples, which were sintered from powder mixture mixed at 100 rpm show former spherical steel particles surrounded by Mg-PSZ clusters. If the powder mixture was mixed at 250 rpm, the Mg-PSZ particles are well embedded in the intensively deformed TRIP steel matrix and the number of ceramic clusters decreases.



Calculations of the densification of the sample during sintering reveal a higher densification rate of the composite powder mixed at 250 rpm. Thus, the densification is almost finished at lower temperatures when the high-energy mixed composite powder compared to the powder mixed at 100 rpm is used for the sintering process. This is in a good agreement with prior results using a comparable composite [10].

The grain growth of the steel grains during sintering is impeded due to the ceramic particles. Furthermore, applying a rotation speed of 250 rpm during powder mixing process causes a grain refinement due to recrystallization during SPS. Thus, the steel grain size of the composites mixed at 250 rpm compared to those mixed at 100 rpm is slightly smaller and decreases with decreasing Mg-PSZ content.

The composites with 5 vol.-% Mg-PSZ exhibit a high ductility and were deformable to 60 % without failure, whereas the composites with 40 vol.-% reveal a significant lower ductility. The 1% offset compressive yield strength ( $\sigma_{1\%}$ ) increases with increasing ceramic content. A further increase of  $\sigma_{1\%}$  is caused by a rotation speed of 250 rpm in the powder mixing process (see Figure 1).



Figure 1. Results of the compression tests.

### 4. Conclusions

The results of the investigation reveal enhanced mechanical properties of the TRIP-steel reinforced with Mg-PSZ due to the increase of the rotation speed from 100 rpm to 250 rpm during ball milling. Applying a higher energy impact causes a clamping between steel and ceramic, a reduction of ceramic clusters and a grain refinement of the steel matrix during sintering. Thus, the compressive strength increases.

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#### Keywords

Metal-matrix composite; high-energy ball-milling; compressive strength; Spark Plasma Sintering