



Article Powder Forging of in Axial and Radial Direction Graded Components of TRIP-Matrix-Composite

Markus Kirschner * D, Sergey Guk D, Rudolf Kawalla and Ulrich Prahl

Institute of Metal Forming, Technische Universität Bergakademie Freiberg, 09599 Freiberg, Germany; sergey.guk@imf.tu-freiberg.de (S.G.); rudolf.kawalla@imf.tu-freiberg.de (R.K.); ulrich.prahl@imf.tu-freiberg.de (U.P.)

* Correspondence: markus.kirschner@imf.tu-freiberg.de; Tel.: +49-3731-39-4188

Abstract: Powder metallurgy is one way of producing complex, graded structures that could allow material systems to be produced with properties tailored to individual applications. However, powder metallurgy requires that the semi-finished products are very similar to the final component. It is much more economical to produce simple semi-finished products and then combine them by powder forging and simultaneous compaction than forming complex components with the desired graded structure. However, it is absolutely necessary that the graded structure of the semi-finished products is maintained during the forming process. In this study, pre-sintered cylindrical semi-finished products, consisting of axially graded as well as radially graded components, were produced by powder forging at 1100 °C. The microstructures, densities and mechanical properties of the final components were investigated to verify the effectiveness of the process route. It was observed that the components formed solid structures after compaction, in which the reinforcing ZrO₂ particles were fully integrated into the transformation-induced plasticity steel matrix.

Keywords: powder forging; TRIP-matrix composite; axial graded structure; radial graded structure; forming

1. Introduction

Constant progress, particularly in the automotive and aerospace industries and in areas such as mechanical and plant engineering, requires materials and components that can withstand ever-increasing loads. Thus, modern materials are expected to have an increasing degree of mechanical, tribological, thermal and chemical resistance. A possible solution for this would be materials with properties specially tailored to the respective application. Such materials are functionally graded materials, as the properties of the components could be adapted locally. Due to their graded structure, such components require functional properties that cannot be achieved by abrupt material transitions. The aim of research in the field of graded materials is to develop materials in which components with different properties adapted to local requirements are optimally combined while avoiding interface problems [1–4].

Powder metallurgy is particularly suitable for the production of such graded components [4–7]. Today the most common method used in powder metallurgy to produce graded materials is layer pressing. These graded structures are built up in layers, and gradually, the concentration or the material properties change within the material system [8,9]. For this purpose, different powder mixtures are filled into the die, layer-by-layer, in the corresponding increments, either parallel for axially graded structures or perpendicular to the pressing direction for radially graded structures. The mixtures are then pressed together and sintered [4,6,8–10]. In this way, however, only a relative density of approx. 90% can be achieved. In order to ensure that the density and thus the property profile is as close as desired to a solid component, a powder forging step following sintering is necessary [10,11]. To ensure a relative density of 100%, a powder forging process with the lateral



Citation: Kirschner, M.; Guk, S.; Kawalla, R.; Prahl, U. Powder Forging of in Axial and Radial Direction Graded Components of TRIP-Matrix-Composite. *Metals* 2021, 11, 378. https://doi.org/10.3390/ met11030378

Academic Editors: Chunfeng Hu and Jose Torralba

Received: 10 December 2020 Accepted: 15 February 2021 Published: 24 February 2021

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.



Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). material flow is used in this paper. In this way, a preform with a simple geometry is formed into a more complex shape of the finished part while simultaneously compacting it [12–15]. The materials most commonly used in powder metallurgy for graded components are ceramics and metals [7,16].

Particle-reinforced metal-matrix composites represent a rather smaller part. Particlereinforced metal matrix composites are suitable for a number of applications due to their improved strength associated with a marginal loss of ductility, the higher stiffness, and the higher wear resistance in comparison to non-reinforced metal [17–19]. In this area, composite materials made of steels with high-energy-absorbing transformation-induced plasticity (TRIP)/twist-induced plasticity in combination with transformable zirconia particles (ZrO₂) show great potential [20–23]. This is because the use of metastable austenitic steels as matrix ensures a deformation-induced increase in both strength and ductility due to the TRIP effect [24,25], while the reinforcement of MgO partially stabilized zirconia (Mg-PSZ) particles increases the strength as well as the wear properties of these composites because Mg-PSZ is capable of undergoing a stress-induced transformation from the tetragonal phase to the monoclinic phase [26–28].

The evaluation of the state of the art of functionally graded materials shows that most of the existing investigations on the production of partially particle-reinforced gradient materials were carried out on axially arranged layers. Very few authors have devoted themselves to the investigation of powder systems with the concentric (radial) layer arrangement, although interesting scientific questions have arisen both for the manufacture of semi-finished products and for the subsequent forming of these semi-finished products in terms of their material flow and compaction properties.

Manolache et al., for example, reports on the production of a radially oriented layered composite using the example of a two-layer cylindrical bushing made of sintered iron and bronze. For this, a tubular preform was first produced from a powder and a resin binder with high porosity. After placing the manufactured preform in a pressing tool and filling the remaining cavities of the die with the second powder, a joint pressing, sintering and calibrating was carried out. Satisfactory part quality was achieved by similar pressing properties of the two powders used [29].

Büdenbender et al., on the other hand, report that a radially oriented layered composite is obtained by joining two cylinders with a different material configuration, which were previously produced by deposition welding. One of these cylinders is a hollow cylinder to accommodate the second cylinder. In a subsequent hot-forming process, the semi-finished product is formed into a hybrid component with complex gear geometry [30].

In Bohr et al., a radially graded component is produced by segregation. For this purpose, an acylindrical matrix was first filled with a binary powder mixture consisting of aluminum and silicon carbide. After filling, vibration and rotation were applied in a certain experimental arrangement to achieve segregation and thus a radial gradient. The entire matrix was then transferred to a press, where the green body was compacted into a component by pressing the powder and then sintering [31].

Baumann uses powder injection molding technology to produce a radially graded component. For this purpose, a binder is added to the binary powder system to create a flowable mass. The material is then injection molded and sintered in several process steps in different layers in order to get a radially graded structure [32].

The aim of this study was to experimentally determine if the forming conditions for the production of axially graded components from a particle-reinforced TRIP-matrix composite using powder forging can be used for the production of axially and radially graded components. The challenge was to ensure that the graded structure was maintained during material crossflow while simultaneously compacting the components without inducing cracks in the sintered parts or fractures during the forming process.

2. Materials and Methods

The investigated material consisted of a gas-atomized, metastable, high-alloyed transformation-induced plasticity (TRIP) steel powder, which was austenitic in structure (with the particle sizes $d_{10} = 8 \ \mu\text{m}$, $d_{50} = 20 \ \mu\text{m}$, and $d_{90} = 127 \ \mu\text{m}$), and a Mg-PSZ ceramic powder (with the particle sizes $d_{10} = 10.6 \ \mu\text{m}$, $d_{50} = 17.8 \ \mu\text{m}$, and $d_{90} = 29.6 \ \mu\text{m}$). The chemical compositions of the TRIP steel (indicated as 16–7–6 TRIP steel) and ZrO₂ ceramic powders are listed in Table 1.

Table 1. Nominal chemical compositions of transformation-induced plasticity (TRIP) steel and Mg-PSZ powders.

TRIP Steel	Fe	С	Cr	Ni	Mn	Si	Ν	Al	S	Мо	Ti
(wt%)	bal.	0.03	16.3	6.6	7.2	1.0	0.09	0.04	< 0.01	< 0.01	< 0.01
Mg-PSZ	ZrO ₂	HfO ₂	MgO	SiO ₂	Al_2O_3	CaO	TiO ₂	Y_2O_3			
(wt%)	bal.	1.85	3.25	0.1	1.58	0.06	0.13	0.13			

The ceramic particle contents were adjusted to 0 *vol*% (0%), 5 *vol*% (5%), 10 *vol*% (10%), 15 *vol*% (15%) or 20 *vol*% (20%). With these different contents, axially and radially graded structures were produced, as shown in Figure 1. For this, a hot pressing was performed to produces semi-finished cylinders with a diameter of 80 mm and a length of 50 mm. The target relative density was set to 90% to simulate the density after sintering in industrial applications. The hot pressed semi-finished products were manufactured at the Fraunhofer Institute for Ceramic Technologies and Systems (IKTS, Dresden, Germany). The arrangement of the layers and reinforcing particles were chosen to resemble a hub or a gear wheel. This ensured that the simulated manufacturing process of the components was as close as possible to the actual manufacturing of the industry.



Figure 1. Types of specimens fabricated.

By subsequent powder forging, the specimens were formed and compacted to 100% relative density. The specimens were formed using a universal forming press at a specimen temperature of 1100 °C (after being heated for 3 h in a protective gas atmosphere to simulate the process after the sintering) and a die temperature of 200 °C. This happened in order to simulate the powder forging process in the industry. Boron nitride was used as the lubricant. The holding time was 10 s, and the pressing force was up to 5 MN. The produced component had a cylindrical form with a diameter of 120 mm and length up to 17 mm. A total of five specimens were formed for each condition. The geometry of the die developed for this purpose was derived from model tests performed using the Gleeble HDS-V40



system (Dynamic Systems Inc., Poestenkill, NY, USA) and is shown in Figure 2. Further information can be found elsewhere in the literature [33–36].

Figure 2. Schematic of the die.

The relative density was determined based on hydrostatic weight measurements, which were performed in accordance with the DIN 53217 standard. Mini tensile tests were performed at room temperature according to the DIN EN ISO 6892-1 standard. The repetition frequency was five times per state, and the specimens were taken parallel to the functional grading direction. For the numerical simulation, a rotationally symmetric FE model was set up using "Abaqus/StandardTM 6.14 Explicit Environment" (Dassault Systems, Vélizy-Villacoublay, France). Light microscopy images and the image analysis of the specimens were obtained by using a VHX-S660E system (Keyence, Neu-Isenburg, Germany) and its software while scanning electron microscopy (SEM) was performed on a GeminiSEM 2 system at 10 kV (Carl Zeiss, Oberkochen, Germany). The microstructures of the specimens, including their phase composition and the characteristics of the zirconia/steel interface, were analyzed through an EDS attachment for energy-dispersive X-ray spectroscopy (Oxford Instruments, Wiesbaden, Germany). For the SEM investigations, the specimens were OP-U polished on a Tegramin 30 (Struers, Willich, Germany). The bulging tests, to investigate the material flow and the flow behavior of the different structures by bulging the material under pressure load, were performed on a hot forming simulator at forging temperature. Theoretically, the flow behavior can be used to set a specific end structure of the functionally graded material by specific adjustment of defined material flows while powder forging. For this purpose, the specimens were placed in a cup with a viewer and then heated up to forging temperature. This was followed by the forming process. The in situ measurement of the specimen was carried out with an LLT2900-50/BL (Micro-Epsilon, Ortenburg, Germany).

3. Results

The semi-finished products were cylindrical with a diameter of 80 mm and length of 50 mm. At the beginning of the investigations, bulging tests were performed in order to obtain information about the material flow and the flow behavior of the different structures by bulging the material under pressure load, see Figure 3. It should be noted that Figure 3 represents a snapshot during the forming process.



Figure 3. Bulging behavior of the different graded specimen types at the same way of deformation of about 10 mm.

It can be seen that the axially graded specimen quickly began to bulge at one end of the specimen. The radially graded specimens, on the other hand, reacted differently, as they started to bulge in the middle of the specimen. It can also be seen that the radially graded specimen (1) was already more bulging than the radially graded specimen (2).

The resulting data can be used to calculate radial, tangential and comparative stresses according to [37,38] shown in Figure 4.



Figure 4. Axially, radially and tangentially stresses in the specimens at the same pressure load.

It can be clearly seen that the stresses in the respective graded structures were different. This was also true for axial, tangential and radial stress. The axial graded structure shows the highest stress in each case, while the radial graded structure (1) lies below. The radially graded structure (2) had the lowest stress. In addition, the axial stress was highest, the tangential stress below and the radial stress was lowest.

To analyze the behavior of semi-finished products during forming with simultaneous compaction, the semi-finished products were formed. To achieve the highest possible density, different compressive forces for the different specimens were required, as shown in Figure 5.



Figure 5. Compressive forces required for different ZrO₂ contents and the graded structure to achieve full density.

It can be seen clearly that the pressing force of the specimens with an axially graded structure required a lower force than the specimens in the radial state. This can be attributed to the differences in the flow conditions and the properties of the different layers. The reason that the radially graded structure (1) needed a higher force than the radially graded structure (2) can be attributed to the particle distribution itself.

Next, semi-finished products after the powder forging are shown in Figure 6

Particle dis- tribution	Тор	Side	Bottom
Axially graded specimens	5 cm		F
Radially graded specimens (1)	5 cm		
Radially graded specimens (2)	5 cm		

Figure 6. Specimen with an axially and radially graded ZrO₂ distribution.

It can be seen that the surface of the specimen was heavily scaled. However, this did not pose a problem since the scale layer flaked off slightly with small impacts, exposing the pure component. Moreover, the die geometry was well mapped in the component. Furthermore, cracks were only visible on the surface of the radial graded specimen (2). These can be explained by the increased ceramic content in the lateral surface of the specimens of radially graded specimens (2).

These images confirmed that components with axially and radially graded reinforcing particle distributions could be manufactured by powder forging. To further verification, the maximum achieved relative density of each specimen and each layer was measured, as shown in Figure 7. The densities of the individual layers were taken from homogeneous specimens produced in the same way [39] and are used here to illustrate the results.



Figure 7. Maximum achieved relative densities of each specimen and layer after the powder forging process.

It can be clearly seen that the relative density of the specimens decreased as the proportion of ZrO_2 increased, as shown in [39]. Furthermore, it should be noted that the fluctuations in the densities of the radially graded specimens were not more significant than those in the densities of the axially graded specimens. It is clearly visible that the radially graded specimens had a lower density than the axially graded specimen. In addition, the radially graded specimen (2) had a lower density than the radially graded specimen (1).

To investigate the microstructures of the different particle distributions, light microscopy images were taken, the location can be seen in Figure 8, and analyzed with image analysis software, as shown in Figure 9.



Figure 8. Location of the light microscopy images.

The axially graded specimen in Figure 9a shows that the particle distribution was retained during the forming process, and the layer thicknesses were nearly equal, as already stated in [39]. The radially graded specimens did not show this type of habit. The radially graded specimen (1) showed that the ZrO_2 poor layers had deformed well, while the ZrO_2 -rich layers had hardly penetrated into the forming zone of increased material flow, shown in Figure 9b. The radial graded specimen (2) showed that the ZrO_2 poor layers started to flow but were blocked by the ZrO_2 -rich layers, shown in Figure 9c. The reason for this was that the functionally graded structure of the radially graded specimens had not expanded further.



Figure 9. Specimens obtained after subjecting pre-sintered specimens to the forming process: (**a**) overview of an axially graded specimen, (**b**) overview of radially graded specimen 1, and (**c**) overview of the radially graded specimen (2).

To evaluate the interfaces more precisely and to analyze the interfacial reactions, SEM analysis was performed on all specimens. The SEM images show that the ZrO_2 particles were embedded within the steel matrix, as shown in Figure 10a. To visualize the differences in the orientations of the particles and the steel matrix easily these a contrast of the backscattered electrons image was chosen, as shown in Figure 10b. It should be noted that the steel matrix had in all specimens an austenitic microstructure and did not undergo a martensitic transformation during the powder forging process. It must be noticed that the dark spots and areas in Figure 10b are not pores in the matrix. In fact, these domains would look like the interfacial reaction products shown in Figure 10a if the grayscale distribution of Figure 10b were optimized for these phases. The black coloration of these oxide phases results from their low backscatter coefficient, which in turn was due to the lower average atomic number of the compound [40]. Furthermore, only a small monoclinic phase fraction in the particles was visible, as shown in Figure 10b. The monoclinic phase appears brighter in the image than the initial phase because ZrO_2 had a higher effective atomic number without Mg, which diffused during the powder forging process and destabilized ZrO_2 .



Figure 10. SEM images based on backscattered electrons: (a) zirconia particle with interfacial reaction products at the interface with steel matrix (magnification: $5000 \times$) [39], (b) characteristic microstructure of composite (two EDS line scans across the interface are highlighted; magnification: $1500 \times$).



Figure 11. EDS line scans shown in Figure 8b: (a) EDS line scan 1 and (b) EDS line scan 2.

On observing the tensile test specimens, it can be seen that the axially graded specimens broke at the head end, while the radially graded specimens (1) broke almost in the middle. The radially graded specimen (2) broke again at one of the heads ends (see Figure 12); the head-end had in the axially and the radially (2) graded specimens the highest content of ZrO_2 -20%, while the highest ZrO_2 content in a radially graded specimen (1) was in the middle. However, all specimens exhibited a fracture angle of approximately 45°.

This fracture behavior was also evident from the results of the tensile tests, which are shown in Figure 13. It should be noted that the volume amounts of the individual layers are the same for the tensile specimens. Only the sequence of the layers, as well as the phase connection between the layers and the density, resulting from the different material flow during powder forging, influenced the result. The axially graded specimen shows the highest strain and stress parameters. The radially graded specimens exhibited a lower strain. It can also be seen in the radially graded specimens that the particle distribution has an influence on the mechanical properties. While the ZrO₂-rich phases were located in the middle of radial graded structure (1), they were located at the outer edge of the radial graded structure (2). The specimen with the particle-rich phase in the core of the specimen has a higher strain than the specimen with the ZrO₂-rich phase on the outside. In addition, these specimens did exhibit a decrease in yield strength and underwent brittle fractures.



Figure 12. Tensile test specimens with (**a**) axially graded, (**b**) radially graded (1), (**c**) radially graded (2) particle distributions, (**d**) light microscopy images of meridian sections of the axially graded specimen, (**e**) light microscopy images of meridian sections of the radially graded (1) specimen, and (**f**) light microscopy images of meridian sections of radially graded (2) specimen.



Figure 13. Changes in mechanical properties with ZrO_2 distribution.

When looking at the hardness values, it is also noticeable, as in the tensile tests, that the radially graded structure (2) has the lowest mechanical properties as shown in Figure 14. In contrast, the axially graded specimens showed the highest hardness, while the radially graded structure (1) fell between the other two curves. It can also be seen that the hardness of the layers, regardless of the structure of the specimens, increased with increasing ZrO_2 .



Figure 14. Changes in hardness with ZrO₂ distribution.

4. Discussion

4.1. Bulging Tests

The reason that the axially graded specimen already began to bulge at one end was due to the predominant hydrostatic compressive stress components in each layer. The hydrostatic compressive stress component decreased with increasing ZrO₂ content, as shown in Figure 15. For a more detailed discussion of the dependencies of the hydrostatic stresses, see [41].



Figure 15. Calculated hydrostatic stresses in each layer of the specimens at the same pressure load [41].

This was caused by the reinforcing particles. This was because they generated a stress field in the matrix. This stress field is opposite to the hydrostatic stress field and reduces formability. This is because the stresses in the matrix caused by the reinforcing particles inhibited the flow of material and material flow occurred later in particle-rich phases than in particle-poor layers [42–44].

The fact that this phenomenon did not occur in radially graded specimens is due to their structure. Because the layers are parallel to the pressing force, all layers must start flowing at the same time. This resulted in a bulging in the middle of the specimen, as described in the literature. The axially graded specimen behaved as already described and predicted in [35]. Thus, the measurements in Figure 3 validate the assumptions in [35].

In Figure 3, one can also see that the radially graded specimen (1) was already bulkier than the radially graded specimen (2). This can also be explained by the structure of the specimens. Since the ZrO_2 -richest layer is on the outside, this layer has a higher proportion of the total volume than any of the other layers. Therefore, the whole specimen started to bulge only when the layer with 20% ZrO_2 started to flow. Since the layer with 20%, ZrO_2

resisted the deformation the most, as described above and in Figure 14, and the proportion of 20% ZrO₂ was higher in relation to the other specimens, the bulge started later.

The reason why the axially graded structure and the radially graded structure (1) had higher stresses than the radially graded structures (2) can be explained by the internal stresses due to the reinforcing particles. Since the amount of 20% ZrO_2 was higher in comparison to the other specimens due to the construction of the structure, the internal stress field was larger than in the other structures. Since this internal stress field was opposite to the other stresses, it reduced them [42–44].

4.2. Compressive Forces

The fact that the specimens with a radially graded structure required a higher force than those for the specimens the axially graded specimens can be attributed to the differences in the properties and flow conditions of the individual layers. The reason that the radially graded structure needed a higher force than the axially graded distribution can be attributed to the particle distribution itself [39]. This is mainly due to the fact that radially graded specimens do not bulge out from the beginning like axially graded specimens, as described in [35]. This is because axially graded specimens will first deform the ZrO₂ poorest layer, and afterward, the ZrO₂-richer layers will follow because the ZrO₂ poor layers have a lower pressing force for complete compaction than the ZrO₂-rich layers (see Figure 5). In addition, the ZrO₂ poor layers were subject to more stress than the ZrO₂-rich layers, as can be seen in Figure 16. This ensured a material flow from the beginning. This is not the case with the radially graded specimens. Because of their structure, all layers must start with the material flow at the same time, which increases the force required.



Figure 16. Pores generated within the matrix of the 20% ZrO₂ layer owing to particle cluster formation [39].

The fact that the force of the radial graded specimen (2) was higher than that of the radial graded (1) could be explained by the amount of ZrO_2 -rich layers. Since the ZrO_2 -richest layer was on the outside, this layer had a higher proportion of the total volume than any of the other layers. Therefore, more force was needed to transform the specimen because the ZrO_2 -rich layers generate a larger stress field, which was opposite to the hydrostatic and deviating stress component. This was confirmed by the poor formability of the edge layers of the radially graded specimen (2) in Figure 4 and the bulging behavior in Figure 3. Since the hydrostatic and deviating stresses in the ZrO_2 -rich layers were smaller than in the ZrO_2 poor layers, the formability was also worse, as already seen in Figure 15 and discussed in [33,36].

4.3. Relative Density

The reason for the different relative densities of the individual layers was due to hydrostatic stresses. Because the reduced formability and the resulting reduced material flow led to different compaction in each layer, as discussed in detail in [33].

However, despite the measurement error, the specimens did not achieve a relative density of 100%. The reason for this was the reinforcing particles themselves. A higher number of reinforcing particles increased the probability of cluster formation in the specimen, as shown in Figure 16. It could be observed how one of the reinforcing particles was

exposed and had a large pore around it, while the other exposed particles were completely enclosed within the matrix.

That the relative density was higher in the axially graded specimens could be explained by the structure of the specimens. While in the case of the axially graded specimens, the forming and compacting of the layers took place one after the other, as described in [33]. In the case of the radially graded structure, it took place simultaneously, as can be seen in the simulations in Figure 17.



Figure 17. Simulation of the forming of the specific graded structures.

It can be seen that with the same strain, individual layers of the axially graded specimens were already formed and hence compacted. This continued layer-by-layer until the whole specimen was compacted [33]. In the radially graded specimens (1), forming started at the outer edge and proceeded to the center. Therefore, it was not possible to condense all spots equally. Especially the edge areas in the middle of the specimen were not be compacted due to the lack of stress and the high resistance offered by the 20% ZrO₂ layer, which was why the relative final density was lower in this structure than in the axially graded specimen. This was because it was not possible to introduce an adequate hydrostatic and deviating stress component in all areas of the specimen to achieve full compaction [45–47]. Since a high hydrostatic stress ratio was required for optimum compaction [46,47], the compaction was lower in the radially graded specimen. Thus, more pores remain. The same was true for radially graded specimens (2). Only with the difference that here it was not the central areas that were not compressed, but the outer mantle surface with high ZrO_2 content. In the radially graded structure (2), the forming process starts in the middle of the specimen and proceeds outwards. Since the stress field generated by the ZrO_2 particles counteracted the forming and compression, the outer surface was not completely compacted. Since the volume fraction of the surface with the ZrO2-rich layer was larger than in the other graded structures, the relative density was lowest in this graded structure.

Because of these aspects, the relative density in the axially graded specimen had a higher relative density than the radially graded specimens, and the radially graded specimen (1) had a higher relative density than the radially graded specimen (2).

4.4. Light Microscopy Imaging

The clusters in Figure 16 can be explained as follows: Clusters increase the local stresses in the matrix. These local stresses are opposing the stress caused by the material flow, which is essential for the compaction and integration of the particles into the matrix. Due to the incipient material flow during the forming process, smaller clusters can be dissolved. However, this material flow was not strong enough to break up larger clusters completely and distribute the particles in the matrix. The material flow in the clusters can even come to a complete halt [38,48]. As a result, complete compaction is no longer possible in this area, which is why the samples with a ZrO₂ content of 20% did not exhibit the desired relative density (see Figure 5).

4.5. SEM Imaging

The oxides in Figure 10 can be explained as follows: Originally, these oxides were formed from the native oxides present on the surfaces of the steel powder particles. Since interfacial reactions take place between the steel matrix and the ZrO2 particles, the oxides were mainly manganese silicated (Mn2SiO4) [49]. The main elements involved in these reactions were Mn, Cr and Si from the steel matrix and MgO from the zirconium oxide particles. Furthermore, Al2O3 impurities from ZrO2 powder production were also involved in the reaction. When the partially stabilized zirconia was forged in the phase field of tetragonal ZrO2 and MgO [50], at 1100 °C, the solid zirconia solution decomposed, and the MgO diffused to the interface. Due to this destabilization process, only small monoclinic zirconia crystallites were generated at the edges of the reinforcing zirconia particles, while the rest of the particles remain in the initial phase [51]. This means that the transformability of the reinforcing ZrO2 phase and the TRIP effect of the matrix was maintained even after powder forging.

4.6. Tensile Tests

The graded specimens broke during the tensile tests on the ZrO_2 -rich phases, as can be seen from the meridian sections of the specimens. It can be clearly observed that the specimens did not crack at the interfaces between the individual layers but above the phase boundary between the 15% and 20% ZrO_2 layers. The reason for this is the particle distribution. Therefore, it can be assumed that the failure at these locations was caused by the pores remaining in the clusters, as discussed above. These pores acted as crack initiation sites due to the notch effect [41,52,53]. Hereafter, the cracks migrated from one reinforcing particle to the next, which caused the cracks to propagate very quickly due to the small particle spacing in ZrO_2 -rich phases, as described in the literature [54–58]. This is the reason brittle fracturing was observed. This can be confirmed from the image given in Figure 18, which shows the propagation path of a crack.



Figure 18. Crack propagation in the 20% ZrO_2 matrix (magnification: 1500×) [39].

In addition, it can be seen that the particle distribution had an influence on the mechanical properties of the radially graded specimens. While the ZrO_2 -rich phases were located in the middle of radial graded structure (1), they were located at the outer edge of the radial graded structure (2). These ZrO_2 -rich phases represent the weak points in the component, as already shown in Figure 10. Since the structure of radially graded specimen (1) contained a relatively smaller amount of ZrO_2 -rich phase than of radially graded specimen (2), the retention of pores and thus component breakdown was more likely. This could also be seen in the density of the specimens because the density of radially graded specimen (1) was higher than the density of radially graded specimen (2). Due to the lower density, more pores remained in the material, which reduces the mechanical properties of the TRIP-matrix composite.

The observed increase in the hardness with the ZrO_2 content can be explained by the reinforcing particles. The particles counteract the deformation-induced during the hardness test, as reported previously [39].

The reason for the lower hardness of the radially graded specimens was due to the structure of the specimens and the compaction. Because of the structure of the radially graded specimens, the specimens were less dense, as already described above and shown in Figure 7. This caused pores to remain in the specimens. However, the pores and thus the relative density had a significant influence on the mechanical properties of components [59]. This, in turn, led to a lower hardness, as also reported in a previous study [33].

5. Conclusions

In this study, we experimentally determined if the forming conditions for the production of axially graded components from a particle-reinforced metal matrix composite using powder forging can be used for the production of radially graded components. The main conclusions of the study can be summarized as follows:

- The powder forging conditions allow compressing specimens with an axially graded particle distribution and at the same time to obtain a local homogeneous particle distribution;
- (2) The powder forging conditions do not allow under the same parameters as for axially graded specimens in order to compress radially graded specimens and at the same time to obtain a local homogeneous particle distribution;
- (3) With regard to the bonding of the reinforcing particles, the powder forging conditions allow in all cases their complete incorporation within the TRIP steel matrix, resulting in a solid compound;
- (4) The configuration of the radially graded specimen structure has a strong influence on its macroscopical density and mechanical properties;
- (5) The layers with high ceramic particle content were the critical regions of the graded specimens, leading to the crack initiation and propagation there;
- (6) The achieved yield strength shows a dependence on the configuration of the radially graded specimen structure;
- (7) The reinforcing particles generate a stress field, which increases with the increasing content of reinforcing particles, in the matrix, which is opposed to the hydrostatic stress field and inhibits forming and compaction;
- (8) The bulging model of axially graded specimens described and predicted in [35] was experimentally validated. The results showed a very good agreement with experimentally determined values;
- (9) The TRIP effect in the steel matrix as well as the tetragonal structure of ZrO2 remained even after powder forging and forming. Thus, no premature phase transformations that negatively influence the properties of the components occur during the manufacturing process.

Author Contributions: Conceptualization, M.K.; methodology, M.K. and S.G.; formal analysis, M.K.; investigation, M.K.; writing—original draft preparation, M.K.; writing—review and editing, S.G. and U.P.; visualization, M.K.; supervision, S.G., U.P.; project administration, S.G. and R.K.; funding acquisition, R.K. and S.G. All authors have read and agreed to the published version of the manuscript

Funding: This work was financed by the German Research Foundation (Deutsche Forschungsgemeinschaft or DFG) and was a part of the Collaborative Research Center TRIP-Matrix-Composites (SFB 799) Subproject A6 I.

Data Availability Statement: The study did not report any data.

Acknowledgments: The authors gratefully acknowledge the German Research Foundation (DFG) for supporting the Collaborative Research Center TRIP-Matrix-Composites (Project number 54473466–

CRC799 subproject A6). Furthermore, the authors thank the Fraunhofer Institute for Ceramic Technologies and Systems for assistance with the manufacturing of the hot-pressed semi-finished specimens.

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

References

- 1. Birman, V.; Byrd, L.W. Modeling and analysis of functionally graded materials and structures. Appl. Mech. Rev. 2007, 60, 195–216.
- 2. Pourmajidian, M.; Akhlaghi, F. Fabrication and characterization of functionally graded Al/SiCp composites produced by remelting and sedimentation process. *J. Mater. Eng. Perform.* **2013**, *23*, 444–450. [CrossRef]
- 3. Liew, K.M.; Lei, Z.X.; Zhang, L.W. Mechanical analysis of functionally graded carbon nanotube reinforced composites: A review. *Compos. Struct.* **2015**, *120*, 90–97. [CrossRef]
- 4. Raßbach, S. Grundlegende Untersuchungen zum Umformverhalten von Gradientenwerkstof-fen unter Anwendung von Druckformverfahren. Ph.D. Thesis, TU Bergakademie Freiberg, Freiberg, Germany, 2002.
- 5. Kolaska, H. Fortschritte bei der Formgebung in Pulvermetallurgie und Keramik: Vorträge Anlässlich des Symposiums am 28./29; Verband Deutscher Ingenieure: Düsseldorf, Germany, 1991.
- 6. Behrens, B.A.; Vahed, N.; Brand, H. *Pulvermetallurgische Herstellung Gradierter Werkzeugwerkstoffe*, 1st ed.; Leibniz Universität Hannover: Hannover, Germany, 2013.
- 7. Elsner, P. Gradierte Werkstoffeigenschaften: Eine Herausforderung für die Fertigungs-technik. Futur 2005, 14, 16–18.
- Zeming, H.; Ma, J.; Tan, G.E.B. Fabrication and characteristics of alumina—Iron functionally graded materials. J. Alloys Compd. 2009, 468, 815–818.
- 9. Li, J.; Sun, W.; Ao, W.; Gu, K.; Xiao, P. Al2O3—FeCrAl composites and functionally graded materials fabricated by reactive hot pressing. *Compos. Part A Appl. Sci. Manuf.* 2007, *38*, 615–620. [CrossRef]
- 10. Sahu, M.; Dargar, M.K.; Bartaria, V.N. The effect of hot forging and heat treatment on tribologocal properties of Al alloy & nano composites. *Int. J. Sci. Eng. Technol.* **2013**, *76*, 649–661.
- 11. Schiller, W. Neue Konzepte zum Kalibrieren von Sinterbauteilen. Diploma Thesis, Montanuniversität Leoben, Leoben, Austria, 2010.
- 12. Exner, H.E.; Danninger, H. Handbook of Inorganic Chemistry, Metallurgy of Iron: Powder Metallurgy of Steel, 10th ed.; Springer: Berlin/Heidelberg, Germany, 1992.
- 13. Kuhn, H.A.; Lynn, B. *Powder Forging. Powder Metallurgy*; U.S. Department of Energy Office of Scientific and Technical Information: Oak Ridge, TN, USA, 1990; pp. 3–19.
- 14. Vossen, K. Pulverschmieden von gerad- und Schrägverzahnten Zylinderrädern. Ph.D. Thesis, RWTH Aachen, Aachen, Germany, 1987.
- 15. Zapf, G. Handbuch der Fertigungstechnik: Urformen; Carl Hanser Verlag: Munich, Germany, 1981.
- 16. Gillia, O.; Caillens, B. Fabrication of a material with composition gradient for metal/ceramic assembly. *Powder Technol.* **2011**, *208*, 355–366. [CrossRef]
- 17. Miracle, D. Metal matrix composites—From science to technological significance. *Compos. Sci. Technol.* **2005**, *65*, 2526–2540. [CrossRef]
- 18. Kaczmar, J.; Pietrzak, K.; Włosiński, W. The production and application of metal matrix composite materials. *J. Mater. Process. Technol.* **2000**, *106*, 58–67. [CrossRef]
- 19. Chawla, N.; Shen, Y.-L. Mechanical behavior of particle reinforced metal matrix composites. *Adv. Eng. Mater.* **2001**, *3*, 357–370. [CrossRef]
- 20. Weigelt, C.; Jahn, E.; Berek, H.; Aneziris, C.G.; Eckner, R.; Krüger, L. Joining of Zirconia reinforced metal-matrix composites by a ceramics-derived technology. *Adv. Eng. Mater.* **2015**, *17*, 1357–1364. [CrossRef]
- 21. Ehinger, D.; Krüger, L.; Martin, U.; Weigelt, C.; Aneziris, C.G. Buckling and crush resistance of high-density TRIP-steel and TRIP-matrix composite honeycombs to out-of-plane compressive load. *Int. J. Solids Struct.* **2015**, *66*, 207–217. [CrossRef]
- 22. Glage, A.; Weigelt, C.; Räthel, J.; Biermann, H. Fatigue behaviour of hot pressed austenitic TWIP steel and TWIP steel/Mg-PSZ composite materials. *Int. J. Fatigue* **2014**, *65*, 9–17. [CrossRef]
- 23. Biermann, H.; Martin, U.; Aneziris, C.G.; Kolbe, A.; Müller, A.; Schärfl, W.; Herrmann, M. Microstructure and compression strength of novel TRIP-Steel/Mg-PSZ composites. *Adv. Eng. Mater.* **2009**, *11*, 1000–1006. [CrossRef]
- 24. Tamura, I. Deformation-induced martensitic transformation and transformation-induced plasticity in steels. *Met. Sci.* **1982**, *16*, 245–253. [CrossRef]
- 25. Zackay, V.F.; Parker, E.R.; Fahr, D.; Busch, R. The enhancement of ductility in high-strength steels. *ASM Trans. Quart* **1967**, *60*, 252–259.
- Weigelt, C.; Aneziris, C.; Ehinger, D.; Eckner, R.; Krüger, L.; Ullrich, C.; Rafaja, D. Effect of zirconia and aluminium titanate on the mechanical properties of transformation-induced plasticity-matrix composite materials. *J. Compos. Mater.* 2015, 49, 3567–3579. [CrossRef]
- 27. Yingkui, G.U.O.; Yu, Z.H.O.U.; Dongbo, L.I.; Xiaoming, D.U.A.N.; Tingquan, L.E.I. Microstructure and performance of 2Y-PSZ/TRIP steel composites. *J. Mater. Sci. Technol.* **2003**, *19*, 137–140.
- 28. Yu, Z.; Yingkui, G.; Dongbo, L.; Xiaoming, D. Effects of load mode on mechanical properties of ZrO2 (2Y)/TRIP steel composites. *Trans. Nonferr. Met. Soc. China* 2003, *5*, 1086–1091.

- 29. Manolache, V.; Schlieper, G.; Huppmann, W.J. Moderne Formgebungsverfahren: Pulvermetallurgie: Pulvermetallurgische Herstellung von Schichtverbunden; Keramik: Berlin, Germany, 1985; pp. 213–220.
- 30. Behrens, B.-A.; Uhe, J.; Wester, H.; Matthias, T.; Büdenbender, C. FE-based layer design of deposition-welded semi-finished parts for the production of hybrid bevel gear. *Procedia Manuf.* 2020, *47*, 309–314. [CrossRef]
- 31. Behrens, B.-A.; Bohr, D. Manufacturing of functionally graded metal matrix composite materials by segregation. *Int. J. Mater. Res.* **2018**, *109*, 373–380. [CrossRef]
- 32. Baumann, A. Pulverspritzgießen von Metall-Keramik-Verbunden. Diploma Thesis, TU Freiberg, Freiberg, Germany, 2010.
- 33. Kirschner, M.; Guk, S. Beitrag zur Bewertung des Verdichtungsgrades Mittels Visioplatsizität; Werkstoffwoche: Dresden, Germany, 2019.
- 34. Kirschner, M.; Eckner, R.; Guk, S.; Krüger, L.; Kawalla, R.; Prahl, U. Deformation behavior of particle reinforced TRIP Steel/MgPSZ composite at hot working temperatures. *Steel Res. Int.* **2019**, *90*, 1800334. [CrossRef]
- Kirschner, M.; Guk, S.; Kawalla, R.; Prahl, U. Further development of process maps for TRIP matrix composites during powder forging. *Mater. Sci. Forum* 2019, 949, 15–23. [CrossRef]
- Kirschner, M.; Guk, S.; Kawalla, R.; Prahl, U. Austenitic TRIP/TWIP Steels and Steel-Zirconia Composites: Powder Forging of Pre-sintered TRIP-Matrix Composites; Springer Series in Materials Science 298; Biermann, H., Aneziris, C.G., Eds.; Springer: Berlin/Heidelberg, Germany, 2020; Volume 1.
- Narayanasamy, R.; Ramesh, T.; Pandey, K.S. Some aspects of workability studies in cold forging of pure aluminium powder metallurgy compacts. *Mater. Sci. Technol.* 2005, 21, 912–916. [CrossRef]
- 38. Guk, S.; Pranke, K.; Müller, W. Flow curve modelling of an Mg-PSZ reinforced TRIP-matrix-composite. *ISIJ Int.* **2014**, *54*, 2416–2420. [CrossRef]
- 39. Kirschner, M.; Martin, S.; Guk, S.; Prahl, U.; Kawalla, R. Forming complex graded and homogeneous components by joining simple presintered parts of TRIP-matrix composite through powder forging. *Metals* **2020**, *10*, 543. [CrossRef]
- 40. Martin, S.; Berek, H.; Aneziris, C.G.; Martin, U.; Rafaja, D. Pitfalls of local and quantitative phase analysis in partially stabilized zirconia. *J. Appl. Crystallogr.* **2012**, *45*, 1136–1144. [CrossRef]
- 41. Kirschner, M.; Guk, S.; Prahl, U.; Kawalla, R. Compaction of Powder Composites for Constructions with High Wear Resistance Requirements. In Proceedings of the XVI International Forum of Young Researchers, St. Petersburg, FL, USA, 17–19 June 2020.
- 42. Flodin, A.; Andersson, M.; Miedzinski, A. Full density powder metal components through hot isostatic pressing. *Met. Powder Rep.* 2017, 72, 107–110. [CrossRef]
- 43. Gasik, M.; Cherradi, N.; Kawasaki, A. FGM components: PM meets the challenge. Met. Powder Rep. 1996, 12, 28–32.
- 44. Rogowski, D. Verfahren zur Herstellung von Preforms für Metal-Matrix-Composites; European Patent Office: Munich, Germany, 2009.
- 45. Dahl, J.R. Powder forged or C-70 steel? Now the MPIF strikes back. *Met. Powder Rep.* 2005, 60, 14–17.
- 46. Thomson, P. Densification of sintered metal compacts by cold deformation. J. Mech. Work. Technol. 1986, 13, 219–227. [CrossRef]
- 47. Schaub, W.; Stilz, M.; Geiger, R. Umformtechnik—Handbuch für Industrie und Wissenschaft: Band 4: Sonderverfahren, Prozeßsimulation, Werkzeugtechnik, Produktion, 2nd ed.; Springer: Berlin, Germany, 1993.
- 48. Pranke, K.; Guk, S. Material flow in Mg-PSZ particle reinforced TRIP-matrix-composites due to hot-rolling. *Key Eng. Mater.* **2016**, 684, 97–103. [CrossRef]
- 49. Berek, H.; Yanina, A.; Weigelt, C.; Aneziris, C.G. Determination of the phase distribution in sintered TRIP-matrix / Mg-PSZ composites using EBSD. *Steel Res. Int.* 2011, *82*, 1094–1100. [CrossRef]
- 50. Pavlyuchkov, D.; Dilner, D.; Savinykh, G.; Fabrichnaya, O. Phase equilibria in the ZrO2—MgO—MnO x system. *J. Am. Ceram. Soc.* **2016**, *99*, 3136–3145. [CrossRef]
- 51. Martin, S.; Decker, S.; Krüger, L.; Martin, U.; Rafaja, D. Microstructure changes in TRIP Steel/Mg-PSZ composites induced by low compressive deformation. *Adv. Eng. Mater.* 2013, *15*, 600–608. [CrossRef]
- 52. Bokuchava, G.D.; Gorshkova, Y.; Papushkin, I.V.; Guk, S.V.; Kawalla, R. Investigation of plastically deformed TRIP-composites by neutron diffraction and small-angle neutron scattering methods. *J. Surf. Investig. X-ray Synchrotron Neutron Tech.* **2018**, *12*, 227–232. [CrossRef]
- 53. Guk, S.; Milisova, D.; Pranke, K. Influence of deformation conditions on the microstructure and formability of sintered Mg-PSZ reinforced TRIP-matrix-composites. *Key Eng. Mater.* **2016**, *684*, 86–96. [CrossRef]
- 54. Li, C.; Ellyin, F. Short crack growth behavior in a particulate-reinforced aluminum alloy composite. *Met. Mater. Trans. A* **1995**, *26*, 3177–3182. [CrossRef]
- 55. Hochreiter, E.; Panzenböck, M.; Jeglitsch, F. Fatigue properties of particle-reinforced metal-matrix composites. *Int. J. Fatigue* **1993**, 15, 493–499. [CrossRef]
- 56. Seo, B.H.; Kim, J.H.; Park, J.B.; Jung, G.D. Crack propagation characteristics of particulate reinforced composites using digital image correlation. *Mater. Werkst.* 2015, *46*, 387–393. [CrossRef]
- 57. Shang, J.K.; Ritchie, R. On the particle-size dependence of fatigue-crack propagation thresholds in SiC-particulate-reinforced aluminum-alloy composites: Role of crack closure and crack trapping. *Acta Met.* **1989**, *37*, 2267–2278. [CrossRef]
- 58. Wang, Z.; Zhang, R.J. Microscopic characteristics of fatigue crack propagation in aluminum alloy based particulate reinforced metal matrix composites. *Acta Met. Mater.* **1994**, *42*, 1433–1445. [CrossRef]
- 59. Vahed, N. *Einfluss des Materialflusses auf die Verdichtung beim Sinterschmieden*; Umformtechnik: Verlag Meisenbach GmbH: Bamberg, Germany, 2014; pp. 2–11.