Chapter 6
Understanding of Processing, Microstructure and Property Correlations During Different Sintering Treatments of TRIP-Matrix-Composites

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Abstract This chapter presents scientifically robust results on the sintering behavior of transformation-induced plasticity (TRIP)-matrix composites based on different consolidation processes, such as conventional sintering, resistance sintering and hot pressing. The correlation of the processing parameters to the adjusted properties, such as the density, porosity, grain size, phase composition and mechanical properties is discussed. The theoretical modeling, including validation with experiments, enabled us to describe, understand and quantitatively optimize the sintering process.

6.1 Introduction

Powder metallurgy (PM) is an attractive production route for conventional metal matrix composites (MMCs) because it offers a wide range of reinforcing particle size amounts and distributions in the matrix material. The additional advantages of PM processes are that the grain growth is lower than that from the ingot metallurgy route, and the reaction between the matrix and reinforcing particles can be minimized by using solid-phase sintering processes. This route is most commonly used in the case of composite materials with steel or other high-performance materials as a matrix that cannot be produced by means of the conventional ingot metallurgy route due to the high processing temperatures, different melting points or different densities of the composite components. Conventional PM production normally consists of three steps: blending the metal and ceramic powders, compacting and sintering. In some cases, a sizing step follows the sintering step. During the processing, such as blending and compacting of the powder particles and pores inside the green compact are adjusted, consolidation processes, such as conventional or conductive sintering, hot pressing (HP) or hot isostatic pressing (HIP), complete the final compaction of the composite material. They differ in the type of heating that occurs (radiative or
conductive heating) and whether additional consolidation acceleration occurs due to the presence an external compressive load (conductive sintering, HP and HIP).

In the past, the described advantages of PM instigated many investigations into MMCs that dealt with the optimization of the sintering processes. In several studies, conventionally sintered composite materials, such as functionally gradient materials (FGMs), that were based on stainless steel with a ceramic component, namely, ZrO$_2$, were investigated [1–4]. The results showed significant residual stresses in the consolidated composite material that resulted in a high concentration of microcracks after the completion of all manufacturing steps. These stresses can be attributed to a difference in the thermal expansion coefficients between the ceramic and steel and the volume expansion during phase transformation. However, an optimal selection of sintering conditions that considers the layer structure leads to good interface quality. For example, a 4-layer FGM extruded in the form of a tube, consisting of a 316L steel matrix and yttrium partially stabilized ZrO$_2$ (PSZ), was produced after sintering at 1350 °C for 1.5 h with a good layer interface quality [5]. The good interface formation in composite materials is based on a chemical reaction at the phase interface, which can cause destabilization of the tetragonal(t) phase of ZrO$_2$ [6]. A variation of the sintering atmosphere showed that while nitrogen positively influenced the mechanical properties of the sintered composite materials, good corrosion properties were achieved by sintering in a vacuum or hydrogen [7].

The consolidation of powders by conductive sintering can be based on different technologies, such as spark plasma sintering (SPS), pulsed electric current sintering (PECS), pulse discharge sintering (PDS), plasma activated sintering (PAS), and field activated sintering technique (FAST). The resistance sintering used in this research is characterized by a direct conversion of electrical energy into heat in the sintered material by applying an electrical voltage to the sample. In [8], a sintering progress was investigated on chromium powder $\Pi_{\chi_1}$ in the temperature range from 1300 to 1500 °C and for sintering times that reached 5 min. The results showed that at the beginning of the isothermal holding time, the effect of the electric current dominated, after which small pores were distributed in the microstructure. The increasing holding time at constant temperature caused low coagulation and enlargement of the pores as well as an increase in the microscopic stresses, which was explained by the action of Laplace forces. Similar investigations of iron powder showed that the densification of a sample to 95% of its relative density when it had dimensions of $\varnothing 12.8$ mm × 3.6 mm was possible by heating at 600 K/s to a sintering temperature of 800 °C and with a sintering time of 6 min [9]. The tests were carried out with a relatively low voltage of 3–10 V and with high current values above 10 kA. The authors indicated that fast densification of the material occurred during the heating phase since at this time, the elevated resistance caused a high Joule heating effect. Furthermore, a theoretical prediction of the influence of the composition of the conductive and nonconductive phases on the physical-mechanical properties of the composite materials was carried out [10]. Theoretical modeling that included experimental validation showed that the limit of percolation (the limiting value of the conductor content at which the conductivity becomes zero) depends on the ratio of the particle sizes of the conducting and the insulating phases. As long as the particle size of the
conducting phase decreases compared to that of the insulating phase, the percolation limit decreases (conductivity can also be achieved with a small content of the conducting phase). In other cases, the percolation limit increases (a small content of the insulating phase leads to the composite material becoming insulating).

Many investigations and broad experience from industrial practice have proven that an externally applied pressure accelerates the sintering process of a porous body. The influence of the pressure on the sintering kinetics at different pressures and temperatures results from the different deformation mechanisms in the porous sintered material, which have been described in detail in Geguzin [11]. The advantages of processes that apply pressure include the manufacturing of dense materials with lower sintering temperatures and shorter sintering times than those of conventional sintering processes; this results in sintered parts with an increased strength that quite often do not require sintering aids. While a substantial amount of knowledge has already been gained regarding the HP of ceramics, metals and cermets, there are only a few papers regarding HP of steel-ZrO$_2$ composites. For example, the properties of a ZrO$_2$(2Y)/transformation-induced plasticity (TRIP)-steel composite produced by HP were investigated depending on the value of applied stress [12]. The powder mixtures were subjected to HP at 1250 °C at 20 MPa for 30 min. The results indicated that the tensile strength and the modulus of elasticity of the composite material decreased under static loading with increasing ZrO$_2$ content up to 35 vol% due to weak bonding at the ZrO$_2$–ZrO$_2$ and ZrO$_2$-TRIP steel phase interfaces. The dynamic yield strength of the composite was greater than that of the static composite due to the induced martensite transformation, which provided improved plasticity and overall formability. The effect of the ZrO$_2$ content on the tensile strength of the composite material at the beginning of dynamic deformation was not evident. The rheological stresses increased with increasing ZrO$_2$ content and deformation in the ZrO$_2$(2Y)/TRIP-steel composites. Nevertheless, the formability and dynamic strength of the composites decreased with decreasing ZrO$_2$ content. Furthermore, manufacturing of FGMs based on tetragonal zirconia polycrystal (TZP) and stainless steel or Ni in the form of SUS304-TZP or Ni-TZP, respectively, could be achieved by HP at 1250 °C for 1 h in a nitrogen atmosphere with 5 K/min heating and cooling rates [13–15]. Finite element modeling of the process indicated that the coefficient of thermal expansion of the composite components was a decisive factor in the production of FGMs free of residual stresses and cracks caused by cooling. Thus, Ni-TZP composites were demonstrated to have better thermal insulating properties than SUS304-TZP.

The postdensification of sintered parts can be achieved by HIP. Unfortunately, there are no data available in the literature about the postdensification of TRIP steel-ZrO$_2$ composites, while sufficient information is available on the HIP behavior of stainless steel powders. The HIP investigations showed that the relative density of AISI 316L samples was more influenced by the HIP temperature than they were by the HIP pressure. The maximum density of 97% was achieved by HIP at 1250 °C and 120 MPa [16]. Furthermore, the compression behavior of AISI 304 steel-based components at various manufacturing parameters was investigated in [17], whereby the manufacturing route consisted of compacting by selective laser sintering (SLS) with
simultaneous cold isostatic pressing (CIP) in the range between 300 and 600 MPa and then sintering in the temperature range between 1250 and 1350 °C for 1 h under a vacuum of $10^{-3}$ Pa. Finally, the components were hot isostatically pressed at 1200 °C at 120 MPa for 1 h. The component manufactured at the maximum CIP pressure and sintering temperature achieved a maximum density of 97% after HIP.

The aim of the present work is the investigation of the densification behavior and final mechanical properties of TRIP-matrix composites depending on the different fabrication processes.

### 6.2 Materials and Methods

Both stainless steel and PSZ powders were used for the investigations. The gas-atomized steel powder was purchased commercially as grade AISI 304 stainless steel (grade 1.4301 stainless steel). Three batches were available, and each was used for one of the three consolidation routes. The batches differed slightly in chemical composition and the resulting delta ferrite fraction (Table 6.1). The particle size varied minimally from batch to batch and was in the range of approximately $d_{10} = 10 \mu m$, $d_{50} = 25 \mu m$ and $d_{90} = 45 \mu m$.

The solidus temperatures of the stainless steel powder batches calculated with the help of the FactSage® program based on the present chemical analysis were 1426 °C (conventional sintering), 1361 °C (resistance sintering) and 1405 °C (HP).

ZrO$_2$ powder was used for the investigations in the partially stabilized ZrO$_2$ (PSZ) form. MgO was applied as a stabilizer. The chemical composition of the powder is shown in Table 6.2. The ceramic particles had an angular shape with a approximate particle size in the range of $d_{10} = 0.2 \mu m$, $d_{50} = 3 \mu m$ and $d_{90} = 22 \mu m$. The phases were distributed among the monoclinic (m), t and cubic (c) phases at nearly equal amounts of 35:32:33%.

The fabrication of MMCs was carried out by conventional and resistant sintering as well as by HP with partial application of the HIP process. The preparation of the powder mixture for all consolidation routes was carried out either by blending for conventional sintering and HP with partial application of the HIP process or by mechanical alloying of the powder mixtures for resistance sintering. Before blending, the volume fraction of ZrO$_2$ was adjusted from 0 to 30 vol%. The inorganic powder mixture was then blended by means of ZrO$_2$ balls stabilized with yttrium for approximately 30 min in a ball mill. Especially for the conventional sintering process, approximately 2.5 mass% of organic binder was added.

The powder mixture was prepared for resistance sintering using a mechanical alloying technique in a vibrating mill with high energy output (attritor) [18]. The grinding duration was approximately 90 s for each 250 g of the initial powder mixture. At the end of the process, the powder mixture was fabricated with an activated surface.

Compacting the green bodies for conventional sintering was carried out by extrusion of the prepared powder mixture. Extruded rods 20 mm in diameter were fabricated using a single-screw extruder with a vacuum chamber in combination with
Table 6.1  Chemical composition of the AISI 304 stainless steel powder for the respective consolidation route, wt%  

| Consolidation route    | C   | Mn | Si | S   | Cr  | Ni | Mo | Ti | Nb  | N   | δ-ferrite |
|------------------------|-----|----|----|-----|-----|----|----|----|-----|-----|-----------|
| Conventional sintering | 0.01| 0.9| 0.2| 0.007| 17.5| 10 | 0.03| 0.004| 0.007| 0.04| 7         |
| Resistance sintering   | 0.08| 0.8| 0.8| 0.014| 21.3| 11.9| –  | –   | –   | 0.25| 9         |
| Hot pressing           | 0.03| 1.4| 0.3| 0.006| 16.7| 8.5 | 0.3 | 0.007| 0.035| 0.126| 18        |
Table 6.2 Chemical composition of ZrO$_2$ powder, wt%  

|         | ZrO$_2$ | Y$_2$O$_3$ | SiO$_2$ | Al$_2$O$_3$ | HfO$_2$ | CaO | MgO | TiO$_2$ | K$_2$O | Na$_2$O | BaO | Fe$_2$O$_3$ |
|---------|---------|------------|---------|-------------|---------|-----|-----|---------|-------|---------|-----|------------|
| wt%     | 96.3    | –          | 0.4     | –           | –       | 0.6 | 2.8 | –       | 0.13  | –       | 0.13| 0.13       |

Fig. 6.1 Parts of the tool for resistance sintering (a) and their locations in the test chamber of the GLEEBLE HDS-V40 (b)

A kneading machine. After the extrusion process, the green compacts were cut into segments of the required length. Afterwards, the water added to the powder mixture before kneading was removed by an air dryer. The relative density of the extruded green bodies was approximately 60%. The subsequent removal of the organic binder occurred in the air at 350 °C for a duration of 5 h. The heating took place up to 100 °C at a rate of 2 K/min and at a rate of 0.2 K/min above 100 °C. The cooling took place at the same rates.

Conventional sintering was performed on extruded and debindered green compacts in an Astro 1100-60100-M1 model refractory metal hot-zone laboratory furnace (Thermal Technology LLC). The accuracy of the temperature measurement was ±4 K over the applied temperature range. According to the furnace manufacturer, the typical temperature homogeneity of the furnace at 1700 °C is ±3 K for 35% and ±6 K for 50% of the furnace chamber volume. The sintering process took place in argon and nitrogen atmospheres (class 5.0) as well as in a vacuum of 10$^{-6}$ mbar. The heating rate was approximately 5 K/min, while the cooling rate was approximately 10 K/min.

A multifunctional GLEEBLE HDS-V40 simulation system (Dynamic Systems Inc.) was used for the resistance sintering. The parts of the specially designed tool for resistance sintering in the GLEEBLE are shown in Fig. 6.1 and consisted of water-cooled base plates, a bottom panel, a ceramic die (Si$_3$N$_4$) with conical inward and outward design features, and a steel press sleeve to reinforce the ceramic die and a punch. To reduce the heat dissipation from the face of the sample in the front through the bottom panel, additional plates of carbon-fiber-reinforced carbon (with a thermal conductivity of 3 Wm$^{-1}$ K$^{-1}$ and specific electrical resistance of 26 $\mu$Ω)
were interposed for thermal insulation. Furthermore, a higher electrical conductivity than that of steel was achieved by using punches fabricated from a material based on a molybdenum alloy with the addition of Ti and Zr. The base plates had current connections but were electrically insulated from the other parts of the GLEEBLE by isolation plates. The powder mixture to be sintered filled the ceramic die before resistance sintering, where the mass of the powder mixture to be sintered was always 250 g. The dimensions of the sintered samples were $\varnothing 50 \text{ mm} \times 18 \text{ mm}$. The ceramic die and tool surfaces were lubricated by hexagonal boron nitride spray before each test to improve the removal of the sintered sample from the tool after the process.

To avoid welding the powder mixture to the bottom panel and the punch as well as carburization of the sample by the carbon-fiber-reinforced carbon plates, a tantalum foil was placed in those areas. After closing the ceramic die with the punch and applying a critical presintering pressure of approximately 50 MPa, the resistance sintering was carried out according to a specified temperature-time cycle with an almost constant sintering pressure of approximately 20 MPa. The temperature of the sample during the sintering process was continuously controlled and adjusted with the help of a thermocouple that was located in the bottom panel and carbon-fiber-reinforced carbon plate so that the heating rate was approximately 50 K/min. Another thermocouple was used for the temperature measurement of the punch and was placed near its working surface. The sample resistance controlled the sintering temperature depending on the $\text{ZrO}_2$ content, the presintering pressure and the sintering pressure as well as the temperature profile. In the course of the resistance sintering tests, the voltage and current values were measured and recorded with an accuracy of $\pm 0.01 \text{ V}$ and $\pm 1 \text{ A}$ using an ALMEMO 2690-8 measuring instrument. The peak values of the voltage and current were approximately 4 V and 6 kA, respectively. Based on the determined values, the electrical resistance of the samples $R$ was calculated according to the following equation:

$$R = \frac{I}{U},$$  \hspace{1cm} (6.1)

where $I$ is an electrical current and $U$ is an electrical voltage.

After the tests, the samples were cooled in the sintering tool before they were removed.

Preliminary tests that directly hot pressed the powder mixtures in the molds (at 1250 °C for 2 h at 30 MPa) resulted in destruction of the graphite molds as a result of the penetration of the mixture into the mold gaps and its interaction with the mold material. For this reason, two methods of mixture precompaction into billets were tested: die cold pressing (CP) of the powder mixture and CIP followed by short-term presintering in a hydrogen atmosphere (Table 6.3). Significant advantages of the first method include the simplicity of the process, high productivity and possibility of mechanization. However, a significant heterogeneity of the billets in terms of the density and inability to produce parts with a complex shape did not allow us to consider this as a universal method. The second method overcomes the disadvantages of the die CP step, but the size inaccuracy of fabricated green compacts and their high surface roughness require the application of additional labor-intensive machining.
Table 6.3  HP consolidation routes

| Method | Steps and their processing parameters |
|--------|---------------------------------------|
| 1st CP | $P = 60$ MPa                           |
|        | $T = 1100, 1200, 1250, 1275$ °C        |
|        | $t = 1$ h                              |
|        | $P = 30$ MPa                           |
| 2nd CIP| $P = 350$ MPa                          |
|        | Presintering                           |
|        | $T = 1100$ °C                          |
|        | $t = 1$ h                              |
|        | $P = 100$ MPa                          |
|        | $H_2$ atmosphere                       |
|        | Post-HIP                               |
|        | $T = 1225$ °C                          |
|        | $t = 1$ h                              |

To avoid the appearance of cracks and stratifications in the billets after CIP, the addition of short-term sintering in a hydrogen atmosphere was carried out, and a relative density of approximately 85% was achieved.

The dimensions of the HP samples were $\varnothing 80$ mm $\times 20$ mm. To remove the residual porosity in the specimens after HP, HIP in an ABRA press apparatus (ABRA Fluid AG) was added and its impact on the microstructure and property evolution was investigated.

The density of the sintered samples was determined in accordance with DIN EN 623-2. The samples for metallographic analysis and mechanical testing were cut from the machined sintered workpieces. Tensile tests were carried out on an AG-100kNNG universal testing machine at room temperature according to DIN EN ISO 6892-1 on cylindrical specimens with dimensions of $\varnothing 4$ mm $\times 20$ mm. The impact toughness of the $55$ mm $\times 10$ mm $\times 10$ mm samples with a V-shaped stress concentration was tested on a RKP450 impact testing machine at room temperature according to DIN EN ISO 148-1. In all cases, samples were obtained in the axial direction for conventionally sintered samples and in the transverse direction for the resistance sintered or hot-pressed samples. The hardness HV10 was determined according to DIN EN ISO 6507-1 on a ZHU250 instrument. The microstructure and fracture surface analysis of the tested samples were carried out by light and scanning electron microscopy. A quantitative evaluation of the micrographs was conducted to determine the pore shape factor $f$ according to Equation:

$$f = 4\pi A / C^2,$$

where $A$ is a grain surface area and $C$ is a circumference of a grain.

The size of the pores and grains was determined with the help of the image processing program Archive4Images. The phase analysis of the ceramics after different consolidation routes was conducted with X-ray diffraction (URD 6 X-ray diffractometer) with an accelerating voltage of 40 kV and current of 30 mA; energy-dispersive X-ray spectroscopy was also conducted.

Based on the experimental results, it was possible to model the sintering behavior with kinetics calculations equations [19] as follows:
\[\rho_{\text{rel}}(t, \vartheta, Z) = (1 + z_1 \cdot Z + z_2 \cdot Z^2) \cdot (\rho_{\text{min}} + (\rho_{\text{max}} - \rho_{\text{min}})) \cdot \left(1 - e^{-\left(\frac{t}{T(\vartheta)}\right)^q}\right)\] 

(6.3)

where \(\rho_{\text{rel}}\) is a relative density, \(t\) is a time, \(\vartheta\) is a temperature, \(Z\) is a \(\text{ZrO}_2\) content, \(z_1\) and \(z_2\) are constants to be determined from the available data. Furthermore,

\[T(\vartheta) = \frac{q}{k} \cdot e^{\frac{Q}{RT(\vartheta)}}\] 

(6.4)

Here, parameters occurring in the equation, such as the Avrami exponent and activation energy, were determined using experimental results and the mathematical-numerical method of nonlinear least square approximation. It was assumed that heating did not have a significant influence on the sintering process, i.e. the assumption of isothermal conditions was used. The modeling approach was sufficient to allow interpolation of the measured values after their successful validation for the purpose of design of optimum technological parameters and thus find an understanding of the changes taking place in the material.

6.3 Results

6.3.1 Conventional Sintering

The results from the investigations showed that the development of the microstructure and pore morphology in the undoped steel matrix (ceramic free) was significantly influenced by the sintering temperature [20, 21]. Particle rearrangement processes took place at a temperature of 1300 °C, which was reflected in the local growth of the pore area (Fig. 6.2). The temperature increase led to the growth of sinter bridges between particles and the associated increase in relative density. This effect is based on the small pores combining with large pores to reduce the surface energy (Ostwald ripening). The final stage of the sintering process was characterized by morphological changes. This involved pore rearrangement due to the ongoing Ostwald ripening phenomenon, including the effect of enclosed gases, as well as the nearly doubled the austenite grain growth. Finally, only closed pores that were isolated from each other were present. The sintering carried out at 1430 °C indicated that the shape of the sample began to change, and this limit was noted.

To achieve advantageous mechanical properties, the favorable sintered microstructure should have, on the one hand, the highest possible value of the shape factor in combination with the lowest possible value of the pore area. On the other hand, minimal grain coarsening should occur. This combination of favorable microstructure properties was achieved at a sintering temperature of 1390 °C.

For the case of a steel matrix doped with ceramic constituents, the sintering behavior was analyzed based on measurements of the electrical resistance of the
specimen. Figure 6.3 presents exemplary results for the composite with 5% ZrO$_2$. The results show that the electrical resistance trend can be divided into three zones for this specimen [22]. In the first zone, the contact between the powder particles was substantially responsible for the electrical resistance of the specimen. The contact area of the powder particles increased during the sintering process, which was reflected in the significant decrease in the electrical resistance. Furthermore, in the first zone, a local region that experienced a resistance increase can be clearly seen in the continuously decreasing electrical resistance near the temperature of 730 °C, where the δ-ferrite phase was dissolved in the austenitic steel matrix according to the phase transformation diagram. Austenitic steel has a specific resistance up to 7 times greater than that of ferritic steel, which caused an increase in the electrical resistance of the composite material. In the second zone, the rate at which the electrical resistance decreased was noticeably reduced. Finally, in the third zone, the resistance passed into the range of pure material resistance. After the sintering process completed and while the specimen cooled, the resistance was slightly reduced due to its temperature dependence.

The scanning electron microscopy (SEM) images, including the chemical element distribution across the interface of the steel matrix doped with ZrO$_2$, showed that there

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**Fig. 6.2** Development of the microstructure and pore morphology in the undoped steel matrix with increasing sintering temperature for a sintering time of 2 h
Fig. 6.3 Electrical resistance of the composite material with 5% ZrO$_2$ during conventional sintering

Fig. 6.4 Bonding between the ceramic and steel matrix for a composite with 2.5% ZrO$_2$ sintered at 1420 °C for 2 h for a single ceramic particle (a) and a ceramic agglomerate (b)

was a good bond between the ceramic particles and the matrix after sintering, while individual particles in the ceramic agglomerates were poorly sintered together due to the sintering temperature being too low for ceramic constituents (Fig. 6.4). The agglomerate formation of the ceramic particles in the cavities of the steel matrix was caused by a significant size difference between the steel and ZrO$_2$ powder particles. Furthermore, an increase in the sintering temperature of up to 1430 °C led to the formation of undesired Al–Mn–Mg-rich areas within the ceramic phase due to diffusion processes through the steel-ceramic interface [23].
The variation of the sintering atmosphere indicated that sintering under vacuum achieved a higher material density than that upon sintering in argon or nitrogen atmospheres. This resulted from practically no gas molecules being present in the pores, which facilitated the rapid shrinkage of the pores. After vacuum sintering, the material had fine pores with a high pore shape factor. However, the disadvantage of vacuum sintering lies in the evaporation of alloying elements from the steel matrix, such as C and Mn, which influenced the austenite stability, the characteristic temperatures of the martensite transformation ($M_s$, $M_f$) and finally the associated mechanical properties.

Furthermore, the negative influence of a high sintering temperature on the phase composition of the ceramic constituent was determined [24]. The exemplary results for the composite material with 5% ZrO$_2$ are shown in Fig. 6.5. It can be seen that increasing the sintering temperature led to an increased content of the undesired m-ZrO$_2$ phase (m-ZrO$_2$) at the expense of the c (c-ZrO$_2$) and t (t-ZrO$_2$) phases, which were reduced.

The sintering of composite materials with varied ZrO$_2$ content showed that the density of the steel matrix doped with 2.5% ZrO$_2$ was similar to that of the undoped steel but with a slight deviation of up to 2% depending on temperature and time, while the material with 5% ZrO$_2$ had a density value similar to that for the composite with 10% ZrO$_2$. The increase in ZrO$_2$ content of up to 10% caused a density increase and associated microstructural developments in the composite materials. The ceramic particles became settled in cavities between the steel particles, which caused, among other things, a decrease in the porosity. An additional increase in the ZrO$_2$ content of up to 30% led to the formation of large ceramic particle agglomerates outside the cavities, which had an unfavorable effect on the densification process and thus on the density of the specimen (Fig. 6.4). Therefore, Fig. 6.6 shows the density of the undoped steel and composite materials with 10 and 30% ZrO$_2$ as a function of the sintering temperature and time. The steel matrix doped with 30% ZrO$_2$ had the lowest
density values herein over the entire investigated time and temperature ranges. After sintering at 1300 °C, its density values were almost as high as those of the undoped steel, regardless of the sintering time. However, a further increase in temperature increased the density difference by up to approximately 13%. The maximum density was 85%. The relative density increase in the steel in the investigated temperature and time ranges was approximately 30%, while that of composite materials was a maximum of 20%. Among the materials investigated over the entire temperature and time ranges herein, the 5 and 10% ZrO₂ composites had the highest relative densities. Thus, the optimal sintering conditions from the point of view of the relative density were determined for the composite materials with a ceramic content of up to 10% ZrO₂. These optimal results were obtained at the temperature of 1420 °C and sintering times of 1–2 h. Under these circumstances, the composite materials with 5 and 10% ZrO₂ after sintering showed a maximum radial shrinkage of approximately 24% compared to that of approximately 18% for the undoped steel.

The results of subsequent modeling were able to show that the applied physical-empirical approach with regard to the ZrO₂ content (6.3) reflected the obtained experimental results sufficiently well (Fig. 6.7). Among the samples herein, the maximum density was observed for a composite with approximately 10–15% ZrO₂. As expected, the density increased with increasing sintering temperature. Figure 6.7b provides an example of how the relative density was influenced by the sintering time for a sintering temperature of 1420 °C and for ZrO₂ contents up to 30%. It is obvious that the ZrO₂ content caused a relative density decrease that reached approximately 13%.

The good validation agreement between the experimental results and the modeling values made it possible to quantify the densification rate of composite materials by a derivation of (6.3) over time. Figure 6.8 shows an exemplary of the densification
Fig. 6.7  Modeling results for the relative density development in relation to the ZrO$_2$ content and the sintering time at sintering temperatures of 1300 °C (a) and 1420 °C (b). The experimental results are marked with dots.

Fig. 6.8  Densification rate of the composite material with 5% ZrO$_2$ as a function of the sintering temperature and time (a) and the ZrO$_2$ content at the sintering temperature of 1420 °C (b).

The densification rate for a composite material with 5% ZrO$_2$ as a function of the sintering temperature and time. Figure 6.8a shows, for example, that the densification rate at the beginning of sintering rapidly decreased from very high values to rates below 0.1%/min within a few minutes, while the dependence on temperature was comparatively very low. The sintering process was thus almost completed after a sintering time of 2 h, and it was confirmed that an optimum was achieved with regard to both the advantageous material properties and fabrication effort.

It can also be seen that the densification rate in the investigated temperature range showed a low dependency on the ZrO$_2$ content (Fig. 6.8b). The composite material with approximately 15% ZrO$_2$ content had the maximum densification rate among the samples herein at the experimentally realized shortest sintering time of 10 min, while the material with 30% ZrO$_2$ showed the lowest densification rate at the same time. Furthermore, the results indicate that the densification rate at sintering times of approximately 1 h and above is practically independent of the ZrO$_2$ content.

Figure 6.9 shows the mechanical properties of the sintered composites as a function of the relative density and ceramic phase content. It is obvious that while the
tensile strength was linearly dependent on the relative density (Fig. 6.9a), the dependence of the total elongation had an exponential relationship (Fig. 6.9b). The increasing in the ceramic content tended to contribute to an increase in the tensile strength, while its large values presented a decrease in the tensile strength due to the increasing tendency of the ceramic to form agglomerates.

The investigation of the fracture surfaces of the undoped tensile specimens revealed that two fracture phenomena occurred depending on the sintering temperature: a prevalent intergranular failure at 1330 and 1350 °C (Fig. 6.10a) and a ductile fracture with a characteristic void structure at 1400 °C (Fig. 6.10b). The microscopic relief of the fracture surfaces in the investigated temperature range essentially depended on the porosity, which was adjusted by the sintering temperature and sintering time. Residual pores and nonmetallic inclusions in the SiO$_2$ and MnO are also visible on the fracture surface since crack propagation through these areas was facilitated.

![Fig. 6.9](image_url) Tensile strength (a) and total elongation development (b) for steel (0Z) and composite materials (from 2.5 to 30Z) after conventional sintering under vacuum

![Fig. 6.10](image_url) The fracture surfaces for steel samples sintered under vacuum for 2 h at sintering temperatures of 1350 °C (a) and 1400 °C (b)
Fig. 6.11 The fracture surfaces for a composite material with 5% ZrO$_2$ sintered under vacuum for 2 h at sintering temperatures of 1400 °C (a) and 1420 °C (b).

The SEM images of the fracture surfaces of the composite material with 5% ZrO$_2$ show a similar failure behavior to that of the undoped steel matrix (Fig. 6.11). Here, single embedded ceramic particles and agglomerates are visible in the austenitic matrix. Pores can be seen in the area between the two phases. In addition, there are visible ZrO$_2$ particles on the fracture surface, like nonmetallic inclusions and pores, that represent the most favorable places for crack initiation and propagation.

The fracture behavior of MMCs with up to 5% ZrO$_2$ after tensile loading was influenced by the relative density and associated strength and ductility. Thus, the MMCs with up to 7% residual porosity showed ductile fracture behavior, which was caused by the void formation mechanism and the coalescence of the micropores. The fracture occurred along the grain boundaries near the residual pores in the steel matrix or near the ZrO$_2$ particles in the MMCs. This effect can be explained by the inhomogeneous distribution of the stress and strain fields due to the mechanical loading of the porous material. The stresses were more likely to be located in the interface region between the grain boundary and the pore than elsewhere since the radius there was minimal. As the sintering temperature increased and the sintering time increased, the material densified to a certain extent while the porosity was eliminated. In the final stage of the sintering process, when only closed pores were present in the microstructure, the intracrystalline pores were formed. The disadvantage of intracrystalline pores is that they can be reduced neither in number nor in size due to the low activation energy for volume diffusion. The advantage is that the intracrystalline pores can be spherical, and spherical pores have a lower stress concentration than that of other shapes, and thus they had a decreased tendency to form notches.
6.3.2 Resistance Sintering

Figure 6.12 shows the temperature and force trends during a stroke-controlled sintering process of the TRIP-matrix composite with 5% ZrO$_2$. The process consisted of three stages [25]. In the first stage, there was a temporary increase in the force for a time up to approximately 400 s, where in the middle of the sample, the temperature increased to 950 °C. This force increase was due to the thermal expansion of the sample and tools. The immediately following second stage is characterized by a reduction in the force with a corresponding large specimen shrinkage and decrease in the porosity. Finally, in the third and final stage, the shrinkage slowed dramatically, which was reflected in the force stabilizing at a constant level. During the entire sintering process, the electrical resistance decreased almost continuously to a few mΩ.

Metallographic analysis for the center of a densely sintered composite material showed an average grain diameter of $d_{50} = 9.6 \mu$m (Fig. 6.13a). Such a fine-grained microstructure resulted from both the mechanical alloying technique and the short residence time at the sintering temperature, where the resistance sintering was dominant. In the high-magnification SEM images, good bonding of the ZrO$_2$ particles to the steel matrix can be seen in addition to individual residual pores on the phase interface (Fig. 6.13b). In contrast, the ceramic particles in the agglomerates demonstrated a poor local bonding quality.

However, the microstructure of sintered samples sectioned across their median showed sufficient inhomogeneities (Fig. 6.14). The highest obtained relative density of approximately 99% was reached in the middle of the sample at the side of the...
Fig. 6.13 Light microscopy image for a composite sample with 5% ZrO₂ sintered at 1100 °C for 11 min (a) and SEM image of the resulting bonding between ZrO₂ particles agglomerate and the steel matrix (b)

Fig. 6.14 Inhomogeneity of the sintered microstructure shown by a quarter cut of a 5% ZrO₂ composite sample. The numbers in the square represent the local relative density measured in %
Fig. 6.15  Temperature evolution in a sintered sample with 5% (a) and 20% ZrO$_2$ (b) as a function of the sintering time for an electrical current value of 290 A

punch (top). These values then decreased in the direction of the side surface of the sample and the water-cooled bottom panel as a result of heat dissipation.

Since the microstructure evolution depended essentially on the temperature distribution in the sample, an analytically closed solution in the form of a Fourier series was searched for by mathematical means and solved numerically for the implemented sintering conditions with regard to the temperature profile [26]. The plot in Fig. 6.15a shows the distribution of the temperature field that arose in a sintered sample with increasing sintering time for an electrical current value of 290 A. The sintered material used here was a mixture of the steel powder with 5% ZrO$_2$ powder. This clearly shows that a temperature gradient of approximately 800 K between the side surface and the center of the sample accumulates after 400 s. When the ZrO$_2$ content increased to 20% and the same electrical current value was maintained, the heating process correspondingly increased due to the increased electrical resistance. This led to a temperature increase of 150 K in the center of the sample (Fig. 6.15b). At the same time, however, the temperature gradient increased, and the microstructure became even more inhomogeneous.

The evolution of the mechanical properties of the resistance sintered samples was similar to that of the conventionally sintered samples. Figure 6.16a shows the local macrohardness measured over the sample cross section as a function of the relevant relative density. The dependence represents a linear relationship. The increase in the nonconductive ZrO$_2$ phase content from 5 to 20% drastically influenced the conductive sintering process since the electrical current only flowed through the steel matrix. Thus, the individual ZrO$_2$ particles in the ceramic agglomerates were not attached to each other. This led to a decrease in the macrohardness. The tensile strengths calculated from the macrohardness values using the correction factor of 3.3 are shown in the plot in Fig. 6.16b. The small differences in the development of the mechanical properties for conventional and resistance sintered specimens were due, on the one hand, to the chemical composition of the initial powders and; on the
Fig. 6.16 Measured macrohardness (a) and calculated tensile strength (b) for composite materials with different ZrO$_2$ contents after resistance sintering under vacuum.

Fig. 6.17 Fracture surface of a composite sample with 5% ZrO$_2$ sintered at 1100 °C for 11 min.

other hand, they were due to the different thickness of the surface oxide layer on the particles, which was influenced by the mechanical alloying technique since the particle size distribution of the powders was the same.

The fracture surface of a sintered sample with 5% ZrO$_2$ is shown in Fig. 6.17. The fine-grained void structure of the austenite with embedded ZrO$_2$ particles can be observed.
6.3.3 Hot Pressing

Figure 6.18 shows the microstructure of the composites with 5% and 10% ZrO$_2$ as a function of the HP temperature. According to the figure, the microstructure consists of austenite grains (light gray) with pronounced twins, PSZ (dark gray) and residual pores (black). A quantitative evaluation of the microstructure indicated that

![Microstructure images](image-url)

**Fig. 6.18** a, c, e microstructure evolution of composites with 5% and b, d, f 10% ZrO$_2$ after HP by the 2nd method at (a, b) 1100 °C, (c, d) 1200 °C and (e, f) 1250 °C
an increase in the HP temperature led to a decrease in the residual porosity and growth of austenitic grains (Fig. 6.19). For all investigated ceramic contents in the steel matrix, the absence of open pores in the samples was observed beginning at the HP temperature of 1200 °C. While the theoretical density of the undoped steel matrix was only achieved at 1275 °C, the addition of 5% ZrO₂ into the powder mixture had a positive effect on the reduction of the required HP temperature by 25 K. The presence of ceramic components in the mixture also had a significant effect on the reduction of the austenitic grain growth. For example, when 10% ZrO₂ was added, the size of the austenite grains at a HP temperature of 1250 °C was reduced by approximately half compared to that of the undoped steel matrix.

The investigations showed that the same HP conditions for composites with 5% ZrO₂ according to the 1st and 2nd precompaction methods to compress the powder mixture into billets without HIP had a different effect on the size of austenitic grains and final relative density of the finished part (Table 6.4). The presintering operation led to an increase in the relative density of the finished part at the expense of the austenite grain growth, which increased by approximately 25%.

To increase the relative density of the undoped steel samples pressed by the 2nd precompaction method at 1250 and 1275 °C to their theoretical value, they were subjected to a final HIP at 1225 °C at a pressure of 100 MPa. The results of the
Table 6.4  Relative density and average grain size of the composite with 5% ZrO₂, hot pressed at 1250 °C according to the 1st and 2nd precompaction methods that compact the powders without HIP

| Method | ρ<sub>rel</sub> (%) | d (μm) |
|--------|---------------------|--------|
| 1st    | 98.6                | 11.5   |
| 2nd    | 100                 | 14.3   |

![Fig. 6.20](image)

Fig. 6.20  Microstructure development after HP by the 2nd precompaction method at 1250 °C without (a) and with a final HIP operation at 1225 °C (b)

Table 6.5  Influence of the post-HIP operation on the relative density and the average grain diameter of the undoped steel powder mixture after HP by the 2nd HP precompaction method

| HP temperature, °C | After HP | After post-HIP |
|--------------------|----------|----------------|
|                    | ρ<sub>rel</sub> (%) | d (μm) | ρ<sub>rel</sub> (%) | d (μm) |
| 1250               | 98.6     | 16.1          | 100          | 44.1   |
| 1275               | 99.8     | 21.3          | 100          | 48.3   |

Microstructural analysis showed that as a result of an additional thermomechanical treatment, there was significant growth of the austenitic grains (Fig. 6.20). Despite the complete densification of the samples, the grain size more than doubled (Table 6.5). A similar effect was also observed in [27] on the same material.

The analysis of the mechanical properties of hot-pressed composites was based on the already observed linear dependence of the tensile strength and exponential dependence of the total elongation on the relative porosity of the material. As shown in Fig. 6.21, the extrapolation of the results into the area of low relative porosity correlated well with the previous results obtained by conventional and resistance sintering of the composites. The insignificant deviations are due to differences in the initial chemical composition of the powder mixture. A comparison of the mechanical properties of the composites with 5 and 10% ZrO₂ shows that a material with 10% ZrO₂ shows increased strength values and decreased total elongation.
Fig. 6.21 Influence of the relative density of composites with 0% (0Z), 5% (5Z) and 10% ZrO$_2$ (10Z) on the tensile strength (a) and total elongation (b) after fabrication by HP according to the 2nd method of precompaction

The results of the tensile tests for samples with 5% ZrO$_2$ by the 1st method of precompaction of the powder mixture into billets indicated a total elongation that decreased by 3 times and an insignificant reduction in the strength; the tensile strength, in particular, was reduced by 1.4 times. The mechanical properties of the undoped steel specimens subjected to the final HIP operation showed that the additional technological step led to a reduction in the yield strength by more than 20% and tensile strength by 1.5 and 3% while increasing the total elongation by 6 and 18% in accordance with the HP temperature [28].

The results of the impact toughness measurements of the composites with 5% ZrO$_2$ showed an increase from 9 to 37 J in the investigated range of HP temperatures [29]. At the same time, the composite with 10% ZrO$_2$ remained brittle because its impact toughness values did not exceed 5 J. The results of the impact toughness measurement on the samples fabricated by the 1st precompaction method also showed lower values than that of the 2nd precompaction method over the whole investigated temperature range. For example, the impact toughness values of the composite with 5% ZrO$_2$ showed a decrease of nearly 4 times to 10 J in the case of the 1st method of precompaction.

Figure 6.22 shows the fracture surfaces of the hot-pressed samples after tensile testing. The precompaction of the powder mixture was carried out by the 2nd method at the HP temperatures of 1100 and 1250 °C. A common feature of the fracture surfaces for the composite with 5% ZrO$_2$ was the ductile nature of the failure with individual brittle ceramic particles. The higher HP temperature of 1250 °C enabled a high elongation of the sample and the production of inhomogeneous and deep dimples on the ductile fracture surface (Fig. 6.22c). The increase in ZrO$_2$ content led to a decrease in the fraction of ductile fracture over the entire investigated HP temperature range. In the fractures of the composite with 10% ZrO$_2$, finely dimpled and mixed failure areas along the phase boundaries corresponding to low ductility are observed (Fig. 6.22b, d).
Fig. 6.22 Fracture surfaces of the hot-pressed composites with 5% ZrO$_2$ at (a) 1100 °C and (c) 1250 °C. Fracture surfaces of the composites with (b and d) 10% ZrO$_2$ hot pressed by the 2nd precompaction method.

Fig. 6.23 Fracture surfaces of hot-pressed composite with 5% ZrO$_2$ by 1st (a) and 2nd precompaction methods (b).

A comparison of the fracture surfaces for the composite with 5% ZrO$_2$ after HP at 1250 °C showed that the samples precompacted by the 1st method (Fig. 6.23a) are characterized by a mixed failure mode and differ significantly from the ductile dimpled structure of the samples precompacted by the 2nd method (Fig. 6.23b).
Figure 6.24 shows that the distribution of the m-ZrO$_2$ crystallographic phase in a composite with 10\% ZrO$_2$ depended on the HP temperature. The results indicate that the distribution function of the ceramic particles that underwent transformation into the monoclinic phase had an exponential dependence [30]. The increase in the HP temperature led to the displacement of the 20\% quantile toward an increase in the m-ZrO$_2$ content. A similar tendency in the slightly smoothed form is also observed for the 50\% quantile, whose values increased from 79 to 92\% with increasing HP temperature.

### 6.4 Conclusions

In this work, the densification behavior and final mechanical properties of TRIP-matrix composites were investigated. The composites were based on 1.4301 stainless steel powder, which was available in the form of three batches with slight differences in their chemical composition and contained up to 30\% MgO-PSZ. The materials were fabricated using three different PM consolidation processes: conventional sintering, resistance sintering and HP. In the case of resistance sintering, a special tool
was designed and optimized to achieve low heat dissipation from the specimen. The microstructure evolution and material properties along each consolidation route were compared to each other. The experimental analysis of the densification kinetics was based on electrical resistance measurements and microstructure evaluation. It was shown that the chemical composition of the steel matrix had a significant influence on the optimum sintering temperature among the investigated sintering technologies.

Experimental investigations of resistance sintering have shown that increasing the presintering and sintering pressures and decreasing the heating rate result in an elevated density of the material. The sintering residence time for composites with a high ZrO₂ content should be extended compared to those with a low ZrO₂ content since the ZrO₂ content delays the consolidation process.

The investigations on HP showed an almost optimal production route, namely, by CIP, subsequent presintering at 1100 °C for 1 h and then HP. The HIP treatment has proven to be ineffective for the postcompaction of hot-pressed samples.

During conventional sintering, the dependence of the composite density on the sintering temperature and sintering time as well as on the ZrO₂ content was quantified by means of a physical-mathematical approach and experimentally supported modeling. It was shown that the densification rate in the investigated temperature range had a low dependency on the ZrO₂ content. The temperature distribution and evolution in the sintered material during resistance sintering was determined based on the physical-mathematical equations as a function of the electrical current value, the sample geometry and the ZrO₂ content. The modeling provided the basis for the densification kinetics and the local character in the composite specimen volume.

A fine austenite microstructure and higher density and tensile strength was achieved with the optimized HP route (2nd precompaction method) and presented a better combination of properties than those of conventional and resistance sintering. Furthermore, low sintering temperatures and short sintering times made the optimized process favorable. The optimally determined HP parameters are 1250 °C with a holding time of 30 min to 1 h and pressure of 30 MPa.

Furthermore, it was shown that the mechanical properties of the consolidated composites primarily depended on the relative density of the material and the ceramic content in the composite. Here, a linear correlation of the tensile strength and an exponential correlation of the total elongation with the relative density of the material was observed. There was no significant influence of the consolidation technique on these mechanical properties. The insignificant deviations could be explained by differences in the initial chemical composition of the powder mixture. With a particle content of approximately 10–30% ZrO₂, strength increases of up to approximately 40% were found. An increase in the tensile strength was accompanied by a decrease in the total elongation. The obtained dependences partially exceeded the mechanical properties of the particle-reinforced composites with a light metal matrix [31]. However, an increasing ZrO₂ content up to 20% caused early failure of the composite under tensile loading due to the formation of ceramic agglomerates with poor bonding to the ZrO₂ particles. Therefore, the best mechanical properties require sufficient
fineness of the ceramic particles (<10 μm) and a uniform distribution as well as good bonding into the steel matrix and low destabilization of the t-ZrO₂ crystallographic modification.

Finally, it was shown that the content of m-ZrO₂ increased with increasing sintering temperature. The increase in the impurities in the ceramic powder caused a greater initial destabilization of the metastable t-ZrO₂ crystallographic phase. In contrast, the influence of the PM consolidation processes investigated in this research on the m-ZrO₂ content had a secondary effect.

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31. Manufacturer specifications: DWA Composite Specialties Inc., Chatsworth, Ca., USA and DACC Dural Aluminium Composites Corp., San Diego, Ca., USA

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