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Effect of different oxide layer shares on the upsetting of titanium aluminide specimens

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Abstract. By ball milling in a low-oxygen atmosphere, it was possible to show that titanium aluminides (TiAl) can be processed into components by pressing and sintering in the same atmosphere. The properties (e.g. hardness and density) that can be realised with established processes such as field-assisted sintering (FAST) or hot isostatic pressing (HIP) were not achieved. Pores in the component are closed by forming processes, which improves the mechanical properties. In this work, powder-metallurgically processed TiAl was hot-formed in a low-oxygen atmosphere. The forging parameters and pre-consolidation were characterised with regard to their effect on the component properties. Force, hardness and porosity measurements as well as metallographic analyses were used to evaluate the process and the resulting specimens. It was found that a pre-consolidation and a higher degree of deformation lead to a lower porosity and a higher hardness.

Introduction

Titanium aluminides (TiAl) are intermetallic materials that have great application potential in the aerospace industry [1]. These materials are characterised by high temperature resistance combined with a low density of 3.9 - 4.2 g/cm³. As a result, titanium aluminides can replace dense nickel-based superalloys (density approx. 8 g/cm³) in engines and thus reduce fuel consumption [2]. The properties of titanium aluminides are mainly determined by the microstructure, which in turn is determined by the manufacturing and processing methods [3].

In order to achieve high chemical and microstructural homogeneity, TiAl alloys are processed by powder metallurgical methods such as field-assisted sintering (FAST) or hot isostatic pressing (HIP) [4]. When processing TiAl powder using the conventional process route, consisting of pressing and sintering, the low ductility and the oxide layers present a challenge. Titanium aluminides have a high affinity for oxygen, which influences the microstructure development and thus the technological properties [5]. In the previously established FAST and HIP processes, the oxide layers are of secondary importance, as the oxide layers are broken up during the process [6, 7].

By ball milling in an oxygen-reduced atmosphere beforehand, oxide-free particle surfaces can be produced and sustained on the TiAl powder. Prior work has shown that TiAl powder can be conventionally pressed and sintered after such a ball-milling process. To sustain the oxide-free particle surfaces, these subsequent steps must however also be carried out under oxygen-free atmosphere. However, these samples exhibited a certain porosity, and the samples also had a lower hardness than comparable material after casting [8]. In principle, the pores in a component can be closed in a forging process, which increases the strength of the component and thus increases the load-bearing capacity of a component. As the strength correlates with the hardness, the hardness also increases as a result of the forming process. This could increase the component properties of

the TiAl sintered parts. However, oxygen also poses a challenge during forming and can lead to material loss [9, 10].

In the present study, the potential of enhancing the properties of TiAl samples produced by pressing and sintering in an oxygen-reduced atmosphere through a subsequent hot forming process in an extremely low-oxygen atmosphere was analysed. In order to analyse the potential of this sinter forging route for TiAl materials, the forging parameters and the prior consolidation are characterised in terms of the effects on the resulting component properties and the process.

Material and methods

The tests were carried out with the titanium aluminide alloy GE48 (composition in % by weight: 59.60 Ti, 33.00 Al, 2.60 Cr and 4.80 Nb). The TiAl powder was produced by inert gas atomisation with argon; the powder had a spherical shape. The oxygen concentration of the powder is 909 ppm_w. 90 % of the powder particles of the initial powder had a particle size below 134 µm. In the cast state according to supplier information, the density of this material is 3.97 g/cm³ and the hardness is 285 HV10 (Gesellschaft für Elektrometallurgie, Nuremberg, Germany).

The study was based on prior work on pressing and sintering of TiAl powder ball-milled under an oxygen-reduced atmosphere, the TiAl powder was processed inside a glovebox (GS Glovebox Systemtechnik GmbH, Malsch, Germany) with an extremely low-oxygen atmosphere. First, it is pre-flushing with argon 5.0 (purity ≥ 99.99 %). Due to the residual oxygen in argon, it is then rinsed with an argon/monosilane mixture (99 % Ar/ 1 % SiH_4). This causes the residual oxygen in the argon 5.0 to react with the monosilane to form silicon dioxide, resulting in a major decrease in the oxygen concentration [11]. The oxygen partial pressure is measured using a sensor box with a lambda probe (L-Probe EM2020, Mesa GmbH, Schmalkalden, Germany). All experiments were carried out at an oxygen partial pressure below 10^{-18} ppm_v [8].

In the glovebox with the low-oxygen atmosphere, the GE48 powder was filled into 2 milling containers (FRITSCH GmbH, Idar-Oberstein, Germany, 250 ml, hardened steel DIN EN 10027-1 X105CrMo17). Each container was filled with 40 g TiAl powder and 345 g steel balls (X105CrMo17) with a diameter of 10 mm. In prior work. toluene (C_7H_8) was added to the powder [8]. It was shown that the toluene reacts with the TiAl and the MAX-phase Ti_2AlC is formed. In order to eliminate the influence of the MAX phase on the forming process within these investigations, no toluene was added to the TiAl powder. The ball milling containers were sealed gas-tight inside the box, so that the oxygen partial pressure was below 10^{-18} ppm_v during the entire ball milling process. The ball milling process was carried out with the planetary ball mill Pulverisette 5/4 (FRITSCH GmbH, Idar-Oberstein, Germany). In prior work the active ball milling time was 4 min with a milling speed of 300 1/min. This was followed by an interruption of 4 min. In contrast to prior work with toluene, this cycle was carried out a total of four times to ensure that the TiAl powder does not agglomerate [8, 12].

After the ball milling process, the ball milling containers were reopened inside the glovebox with the low-oxygen atmosphere. Green compacts were then produced from the TiAl powder. Compaction was carried out by coaxial pressing on the manually driven hydraulic press (MSE Teknoloji. Osb/Gebze/Kocaeli, Turkey) inside the glovebox. This hydraulic press is located inside the glovebox in a separate gas-lockable box. In each case, 20 g of TiAl powder was filled into the die and compacted at 800 MPa, the samples had a diameter of 20 mm. These green compacts were then filled into capsules inside the glovebox and sealed gas-tight. The capsules consist of three parts, with 2 lids and a hollow cylinder, the inner diameter of the hollow cylinder and the outer diameter of the lids were 20 mm. A press fit of H6/n6 according to DIN ISO 2768 between hollow cylinder and lid ensures a gas-tight seal. To seal the capsules with the TiAl, the separate box with the hydraulic press (MSE Teknoloji) was closed and a negative pressure of less than 50 mbar was created inside using a vacuum pump. This is necessary to ensure that there are no gas pockets in the capsule which can cause porosity. The lids were then pressed into place using the hydraulic

press. The closure ensures that the following forming outside of the glovebox can also take place in an oxygen-reduced atmosphere. With regard to the choice of material and wall thicknesses, the design of the capsules was based on Wan et al. and Li et al. who have carried out investigations on the forming of cast and encapsulated semi-finished TiAl products [13, 14]. All components were made of 1.4301 steel and the capsules had a length of 32 mm and an outer diameter of 30 mm. To investigate the effects of the oxide-free surfaces during forming, 20 g of unground and therefore oxidized GE48 powder was filled into capsules as a reference. As it is not possible to produce green compacts from untreated TiAl powder by die pressing due to the oxide layers, the powder was filled into the capsules without compaction [8].

Half of the capsules with ground TiAl powder and half of the capsules with unground TiAl powder were then subjected to a sintering process to identify the effect of prior consolidation and the interaction with possible further consolidation through the hot forming process. This results in 4 different process routes for this study. This sinter process was carried out in a sintering tube furnace (Thermconcept ROC, Bremen, Germany) at 1150 °C for 60 min at a heating rate of 5 K/min. The temperature selected for the sintering process is based on previous FAST experiments in which a desired near γ microstructure was successfully achieved at this temperature [15]. The furnace is directly connected to the glovebox via a cooled flange to guarantee a transfer under the required low-oxygen atmosphere. The sintering furnace is supplied with the low-oxygen atmosphere of the glovebox by a fan, so that the oxygen partial pressure during sintering was also below 10^{-18} ppm_v. The other half of the samples without sintering are analysed in terms of the potential for a direct powder forging process, investigated by Jiang et al. for nickel-based superalloys [16].

For the hot forming tests, the corresponding samples were heated in an oven at the selected temperature for 60 minutes so that the sample had a homogeneous heat distribution. During the forming process, the force was recorded with the force measuring device (see Figure 1) based on strain gauges. The maximum force was recorded in order to analyse the effect of the different process routes and the forming parameters on the deformation behaviour and mechanisms. The maximum correlates with the highest degree of deformation towards the end of the forming operation and is therefore most significant to characterise forming.

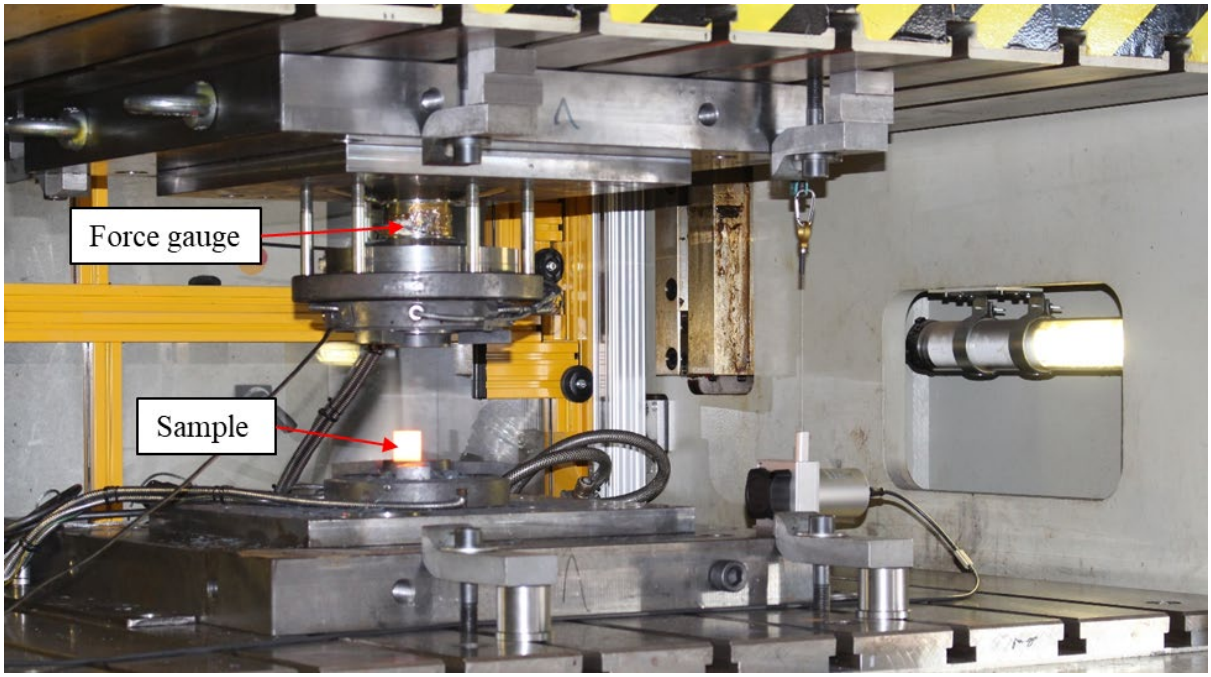

Figure 1. Setup of the forming tests

All samples were hot-formed on a Dunkes HD 250 (S. Dunkes GmbH Maschinenfabrik, Kirchheim unter Teck, Germany) hydraulic press. The forming parameters were varied individually in order to investigate the influence of the forming temperature, pressing speed and the degree of forming on the process and the properties of the samples. To determine the degree of deformation from the initial height h_0 before upsetting and the final height h after upsetting, the following formula is used:

$$\varphi = \ln(h/h_0) \quad (1)$$

The parameters in Table 1 were used in the tests, which were carried out according to a partial factorial test plan. This results in 7 forming processes for each specimen preparation state, resulting in a total of 28 tests for this study. The parameters used are based on previous hot forging tests in which recrystallisation was observed at higher forming speeds and degrees [17]. Due to the gas-tight closure of the capsules, each forming process was carried out in an oxygen-reduced atmosphere.

Table 1. Applied forming parameters

Degree of forming φ	Pressing speed v	Forming temperature U_T
-1.16 [-]	10 [mm/s]	1050 [°C]
-0.76 [-]	30 [mm/s]	1150 [°C]
-0.47 [-]	50 [mm/s]	1250 [°C]

After the forming tests, cross-sectional samples were produced to determine the properties of the titanium aluminide. In order to be able to evaluate the mechanical properties despite the limited specimen dimensions, hardness measurements according to Vickers HV1 were carried out using a Qness Q10A hardness [10]. In the centre of each specimen, 15 hardness measurements in accordance with ISO 6507 were carried out and averaged. A confocal LED microscope (Smartproof 5, ZEISS, Oberkochen, Germany) was used to quantify the porosity in the centre of the sample. The cross-section surface was scanned in an area of 550 µm x 480 µm. To differentiate between roughness due to specimen preparation and depressions in the surface caused by porosity, a tolerance plane of 0.25 µm below the highest peak was applied in accordance to [18]. Any cavity below this plan is defined as porosity. The areas with the indentations were compared with the scanned cross-sectional area. Evaluating the surface porosity ratio of the specimens allows a comparative analysis of the densification behavior of the applied forming processes. In addition, global stitched images of the entire cross-section of the sample were taken with a Leica DM6 light microscope (Leica, Wetzlar, Germany) in order to make statements about the overall microstructural homogeneity of the formed specimens. By applying these analytical methods, to establish a deeper understanding of the correlation between the pre-consolidation of low-oxygen processed powder and the resulting properties.

Results and discussion

Process forces

For a qualitative statement on the influence of pre-consolidation on the process force, the maximum force was measured for each test. The maximum force was reached at the end of the forming process. The results of the maximum force is listed in Table 2 for each test with different specimen conditions and forming parameters. The results show that the influence of the forming parameters on the maximum force during the forming process is greater than the influence of the process route of the TiAl powder. As expected, the process forces increase with lower forming temperature, a higher degree of forming and a higher pressing speed. There is no clear influence of milling and sintering before hot forming on the process forces. The oxide layers, which inhibit particle bonding and thus minimise the strength, do not result in a significant reduction in the

forming forces. Overall, the forces for each forming parameter setup were highest for the ground and sintered variants. This can possibly be caused by the stronger particle cohesion after pressing and sintering and therefore stronger overall material strength which must be overcome in forming.

Table 2. Results of the maximum force measurements

	Reference		**Ball milled**	
	Hot Forming	Sintering and Hot Forming	Hot Forming	Sintering and Hot Forming
φ = -0.76, v = 30 mm/s, U_T = 1050 °C	329 kN	317 kN	322 kN	331 kN
φ = -0.76, v = 30 mm/s, U_T = 1150 °C	247 kN	252 kN	259 kN	275 kN
φ = -0.76, v = 30 mm/s, U_T = 1250 °C	200 kN	208 kN	195 kN	185 kN
φ = -0.47, v = 30 mm/s, U_T = 1250 °C	108 kN	111 kN	122 kN	118 kN
φ = -1.16, v = 30 mm/s, U_T = 1250 °C	381 kN	368 kN	363 kN	384 kN
φ = -0.76, v = 10 mm/s, U_T = 1250 °C	155 kN	151 kN	164 kN	176 kN
φ = -0.76, v = 50 mm/s, U_T = 1250 °C	265 kN	282 kN	259 kN	260 kN

Porosity

Figure 2 shows the optical microscope images of representative samples. In the partial images a) to d), the titanium aluminide (grey) can be seen in the centre and the steel capsule (light grey) at the edge. As in the study by Jiang et al., the light micrographs show that the porosity is inhomogeneous and increases towards the edge of the sample [16]. The increase in black dots towards the edge shows this. This can be led back to local differences in plastic strain, which generally reach a maximum in the centre for cylinder compression tests. The unground variants have a higher porosity over the entire cross-section than the ground variants, see Figure 2 c) and d). It can therefore be concluded that higher green strength can reduce porosity of the finished part. In addition, the oxide layers act as a diffusion barrier during heat treatment, thus favouring porosity [19]. The optical microscope images showed that most of the samples contained cracks. A tendency depending on the process route or the forming parameters cannot be recognised. It can be assumed that the selected pressing speeds are too high. In the investigations by Bolz et. Al., pressing speeds of less than 0.05 mm/s were used to prevent cracking [20]. However, the longer contact time due to the low pressing speed led to an increase in the diffusion layer between the die and the component, which had to be removed afterwards. A diffusion layer between the steel capsule and the titanium aluminide can also be seen in Figure 2 a) and c). This can be seen in the colour transition from the grey titanium aluminide to the light grey steel capsule. As diffusion is temperature-dependent, the diffusion layer is less pronounced at the lower forming temperature, Figure 2 b).

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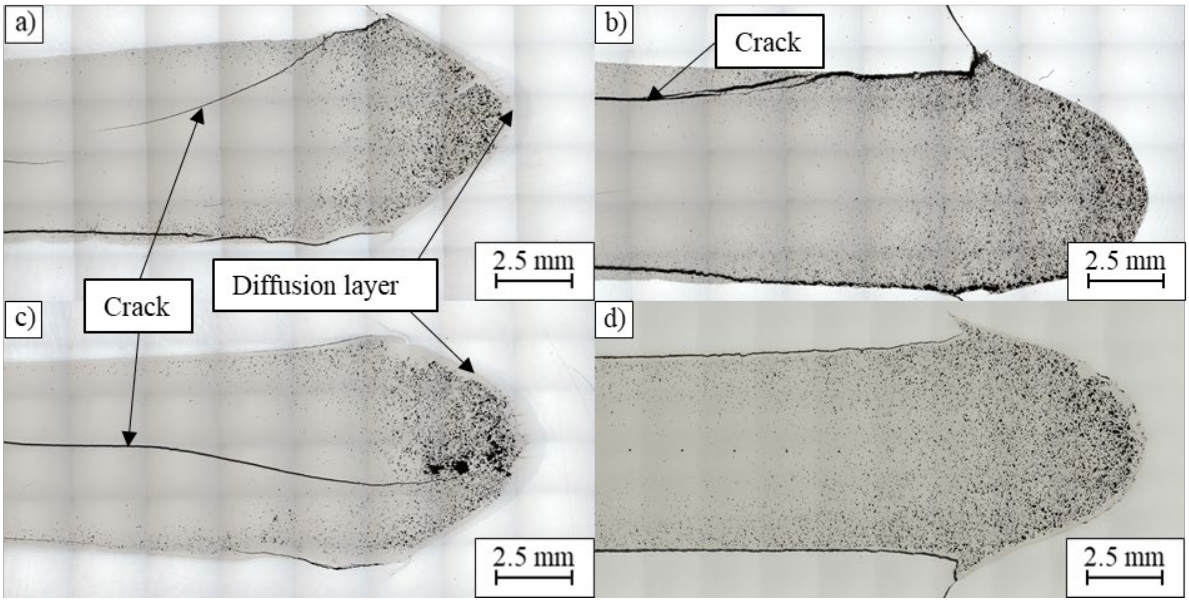

Figure 2. Light micrographs: a) ball milled and sintered, φ= -0.76, v= 30 mm/s, U_T = 1250 °C; b) ball milled and sintered, φ= -0.76, v= 30 mm/s, U_T = 1050 °C; c) ball milled and not sintered, φ= -0.76, v= 30 mm/s, U_T = 1250 °C; d) Reference and not sintered, φ= -0.76, v= 30 mm/s, U_T = 1250 °C

By forming, it is possible to achieve a porosity of less than 0.05 % and thus almost completely seal the material, see Figure 3. However, the results show that different porosities are achieved depending on the selected parameters. When varying the forming temperature, it can be seen that the porosity is highest at 1150 °C. This temperature is close to the eutectic temperature. This temperature is close to the eutectoid temperature (1125 °C) of TiAl, at which a phase transformation takes place [21]. It is assumed that this transformation causes a higher porosity. In addition, the variation of the pressing speed shows that the lowest pressing speed has the highest porosity. It is assumed that the higher heat loss during forming due to the lower speed means that the diffusion processes are not fully completed, so that the porosity is not further reduced. A higher pressing speed is therefore recommended for lower porosity, but this can lead to greater cracking. When varying the degree of deformation, it was found that a degree of deformation of -0.47 leads to high porosity and decreases at higher degrees of deformation. It can be assumed that the higher forming force due to the higher degree of deformation leads to further consolidation of the material and influences the mechanical properties.

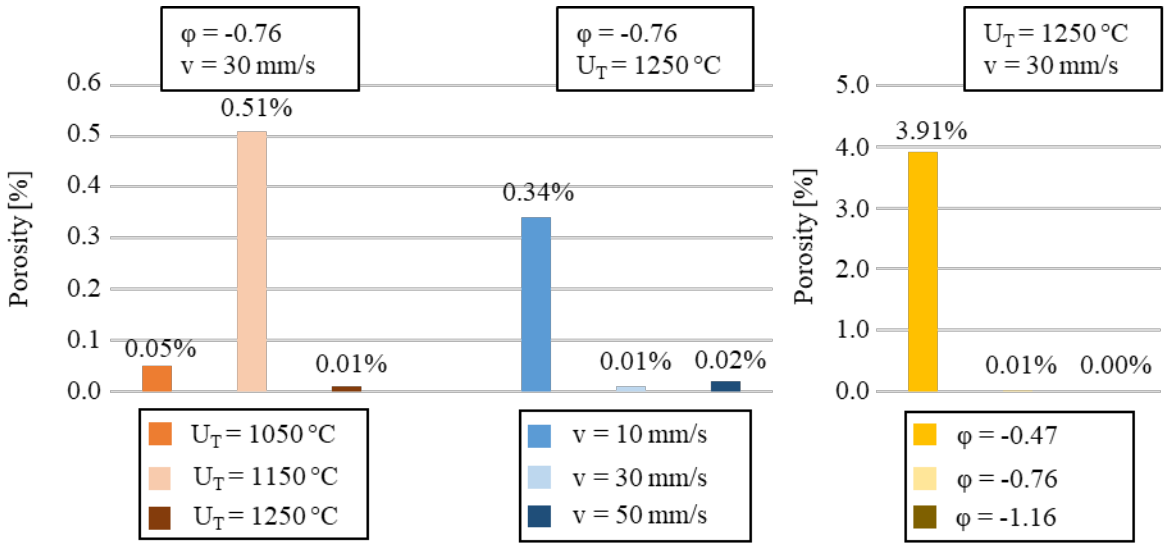

Figure 3. Results of the porosity measurements for ball-milled, sintered specimens after forming

Hardness

The hardness results show that hot forming leads to an increase in hardness. All samples exhibited a significantly higher hardness than the sintered specimens (152 HV1) in the prior study [8]. During the forming process, the pores are closed by the wrought process, which improved the mechanical properties of the TiAl sample [9]. Figure 4 shows an example of the hardness values from a series of tests. It can be seen that the ground variants have a higher hardness than the unground variants. It can be assumed that the oxide-free surfaces created as a result of ball milling in a low-oxygen atmosphere lead to an increase in green strength during pressing due to the improved cold welding. These juvenile surfaces and the increased cold welding favour the diffusion process during sintering, which is why the hardness of the titanium aluminide increases again in the sintered variants. The hardness is further increased with the subsequent forming process. The sintering process before hot forming also leads to an increase in hardness in the untreated samples. In the untreated TiAl samples, the oxide layers hinder diffusion [19].

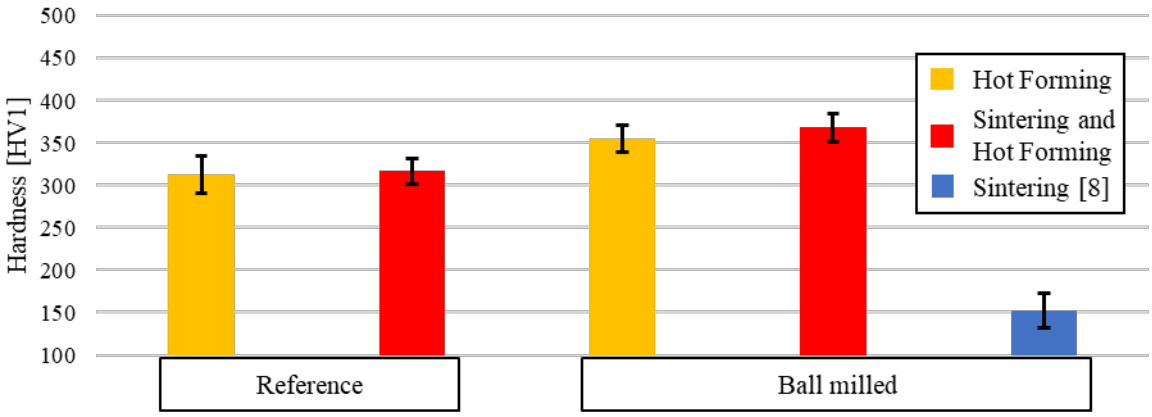

Figure 4. Comparison of hardness values within a test series: φ= -0.76, v= 30 mm/s, U_T= 1150 °C

The results of the milled and sintered variants from the study are shown in Figure 5. When varying the forming parameters, this study shows that the forming temperature has not an effect on the hardness of the samples after forming; the differences are within the standard deviation and therefore not significant. The standard deviation represents the material homogeneity. As the selected forming temperatures are above the recrystallisation temperature, the temperature

differences have no significant influence on the global hardness [22]. However, it can be seen that the standard deviation is highest at the forming temperature of 1150 °C. Thus, as with the porosity measurements, an influence of the phase transformation near the eutectoid temperature (1125 °C) on the homogeneity of the hardness curve can be assumed [21]. When varying the pressing speed, hardness measurements also showed no significant differences; the deviations are within the standard deviation. However, it was found that the standard deviation decreases with increasing forming speed. It is assumed that the lower heat loss due to the higher pressing speed leads to a higher homogeneity of the hardness, as the diffusion processes can take longer. When varying the degree of deformation, it was found that the hardness increases with increasing degree of deformation. By increasing the degree of deformation, the porosity in the TiAl samples decreases as a result of the forging process, which increases the hardness [9]. The hardness of the variant with the highest degree of deformation was 412 HV1, an increase in hardness of over 170 % compared to sintering [8]. Compared to the cast material (285 HV10), the hardness of the moulded sample is also over 40 % higher. It can therefore be concluded that increased pre-consolidation and a higher degree of forming lead to an increase in hardness

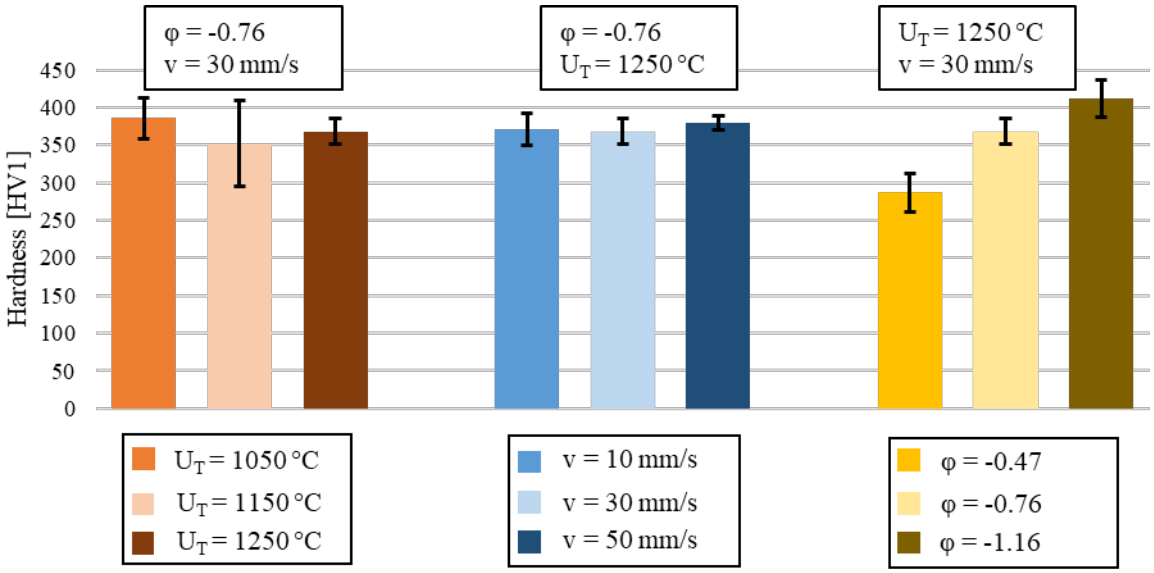

Figure 5. Comparison of hardness values with variation of the forming parameters for ball-milled, sintered specimens after forming

Summary

In this study it is analysed how the properties of titanium aluminide produced by pressing and sintering can be enhanced through a hot forming process in an oxygen-reduced atmosphere. The influence of milling in an oxygen-reduced atmosphere before pressing and sintering and hot forming was analysed. Both the milling of the TiAl powder and the sintering before forming had no significant influence on the maximum forces during forming. The correlation between low porosity and high hardness was also confirmed. Ball milling and sintering as well as a higher degree of forming lead to a higher hardness and a lower porosity over the entire cross-section. Compared to the sintered sample, an increase in hardness of over 170% and compared to the cast material, an increase in hardness of over 40% could be achieved. In addition, the pores could be almost completely closed. However, it was shown that forming close to the eutectoid temperature, a low pressing speed and a low degree of forming lead to a high porosity. In subsequent work, the results should be validated through further volumetric density measurements. The influence of the eutectoid temperature on the density of TiAl will be examined in more detail. Further work is also required to homogenise the density globally throughout the specimens, as the high density

presented here is limited to the centre with the highest plastic strain. The extent to which hardness can be increased and porosity reduced by increasing the degree of forming must be determined in subsequent studies.

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