PM2001
Porous Materials

Investigation of PM Production Route and Characterisation of some Mechanical Properties of Highly Porous Titanium

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Abstract

Some peculiarities of the powder metallurgy route for the production of highly porous titanium have been studied. The requirements of the starting titanium and pore-forming powders were formulated. The compaction behaviour of these powders and their mixtures was investigated. It was found that the pore-forming additive has a lubricating effect. The shrinkage of highly porous samples with respect to the compaction pressure, sintering temperature and time was investigated. The densification of the titanium network was observed as a main reason for the shrinkage. The radial dimensional changes of porous samples were close to the radial shrinkage of pure titanium specimens compacted at the same pressure. This peculiarity allows graded structures to be manufactured consisting of layers with different porosities. Compression tests were performed for the investigation of deformation behaviour and to establish the relationship between the level of stresses and the macroscopic response of porous material. The strong anisotropy of the strength and elasticity characteristics was determined. Additionally, fracture toughness was tested under static loading.

Introduction

Highly porous titanium can be manufactured by the PM route using pore-forming additives [1-3]. This material has good prospects for new biomedical applications as an ultralight prosthesis for extremities. Technology includes preparation of the mixture, containing basic Ti powder and pore-forming additive, its compaction, and removal of the pore-former by heating and subsequent sintering. The realisation of this technology on an industrial scale requires a good understanding of each processing step and knowledge of resulting properties of porous parts. At present there is a lack of information on these features in the literature. Therefore the corresponding experimental investigation was carried out in cooperation between two German Research Centres - Jülich and Geesthacht [4]. The results obtained are the subject of the present paper.

Experimental procedure

It was found that the key factor for the successful realisation of the discussed technology is the right selection of the basic and pore-former powders. The basic (titanium) powder must combine high ductility with irregular particle shape. Then the stable contacts between the powder particles can be achieved by compaction. These contacts should ensure sufficient green strength of the components after removal of the pore-forming additive by low temperature heating. The number of such contacts depends on the size ratio of the pore-former to titanium particles. The pore-former particles must be several times larger than the particles of the basic material. This contributes to increasing the number of titanium to titanium contacts and to decreasing the number of the pore-former to pore-former and pore-former to titanium contacts. The last two contacts disappear after pore-former removal. It is clear that the importance of this rule grows with an increase of the pore-former volume content. Titanium powder < 75 μm (GfE, Germany) and pore-forming powder of 355-500 μm were used in the present investigation. The highest volume content of the pore-forming additive was 75%. Fig.1 illustrates the morphology of Ti and pore-former powders.

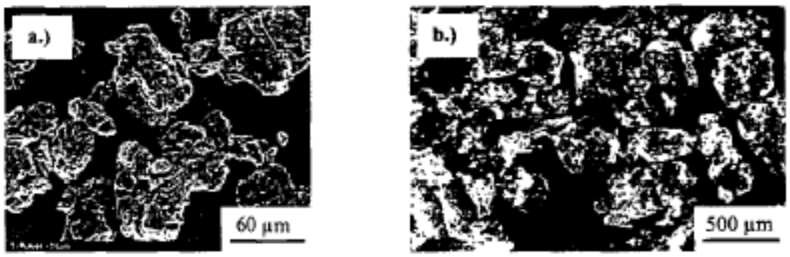


Fig. 1: SEM images of powders used for investigation **a.)** titanium powder **b.)** pore-forming additive.

Compaction and sintering behaviour were investigated for the pure basic titanium powder as well as for its mixtures with additives in ratios of 70:30, 50:50, 30:70 (vol.%). To increase the stability of the mixtures and to prevent segregation, a small amount of the readily evaporating solvent was added to the mixtures. In the investigation of compaction, the pure pore-forming additive was also used. The mixtures were homogenised on a rolling bench for several hours without using grinding balls to avoid destruction of pore-former particles and to keep the impurity level low. Uniaxial compaction was carried out in a pressing die with a diameter of 18 mm at 120, 200, 270, 350 and 420 MPa to investigate the influence of pressure on green density and ejection stress. The height of the compacts was around 15 mm. Additionally, several samples were compacted in a pressing die with a diameter of 60 mm. These samples were used in the study of mechanical properties of porous titanium. The pore-forming agent was removed by thermal treatment below 300°C. To investigate the vacuum sintering of Ti compacts, four temperature cycles were used (1200°C for 1h and 3h as well as 1300°C for 1h and 3h). Axial, radial shrinkage and sintering density were measured to characterise the sintering behaviour of compacted samples. Compression and fracture toughness tests were performed at the GKSS Research Centre to study mechanical characteristics of porous titanium. The compression tests on the rectangular samples (length x width x height 12x12x10 mm^3) were done at room temperature in compaction and perpendicular to the compaction direction. The aim of this investigation was to study the anisotropy of the stress-strain response of the porous titanium. Standard fracture toughness tests were carried out using CT-type (10 mm thick) specimens manufactured by spark-erosion cutting.

Results

Compaction

Fig. 2 illustrates the dependencies of relative density on uniaxial compaction pressure for titanium powder, additive and their mixtures. Due to the high densification capability of the additive, the green density of mixtures was enhanced by increasing the additive content. On the other hand, the ejection stress diminished when the amount of additive was increased. This result indicates the lubricating effect of the pore-former which is similar to the conventional pressing aid. In general, the ejection stress was in the range of 5 - 8 % of compaction pressure.

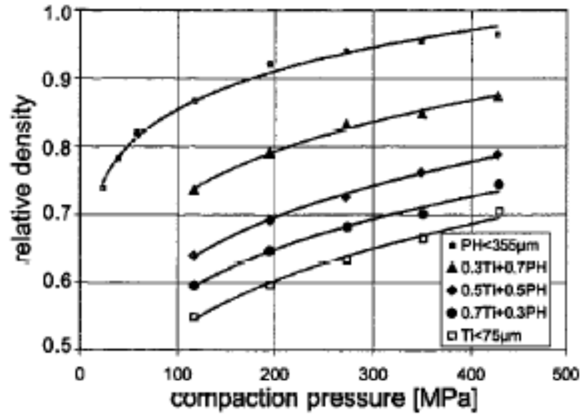
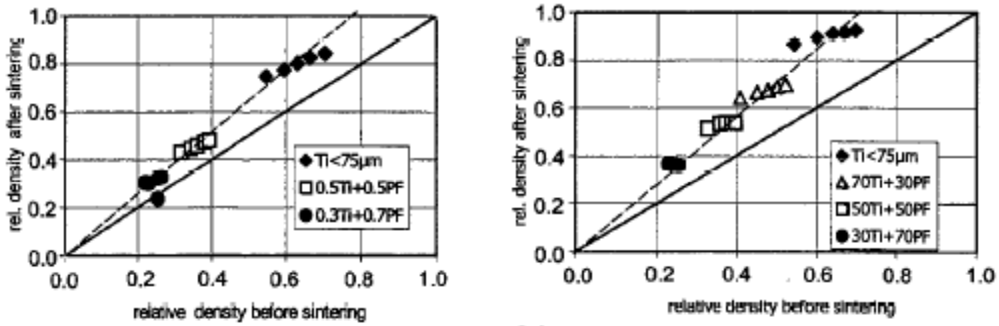


Fig. 2: Relative density vs. pressure by uniaxial compaction of various titanium additive mixtures (PH means pore-forming additive).

Sintering

To clarify the influence of compaction pressure and sintering parameters on the remaining microporosity, the pure titanium powder and the three mixtures mentioned above were compacted at different pressures and sintered in vacuum. The shrinkage and the density increments were determined. Fig. 3 indicates that the density increase caused by sintering is relatively small. Obviously, it depends only marginally on the content of pore-forming additive.



a.) **b.)**

Fig 3: Density increase by various sintering cycles: **a.)** 1200°C, 1h; **b.)** 1300°C, 3h. Five samples of each parameter set are shown.

This observation can be explained by assuming, that densification during sintering mainly occurs as a result of shrinkage of small pores in the titanium network. This idea was verified by the investigation of the microstructure development at various sintering temperatures. The results are presented in Fig. 4. Here the macroporosity caused by the pore-forming additive was kept constant

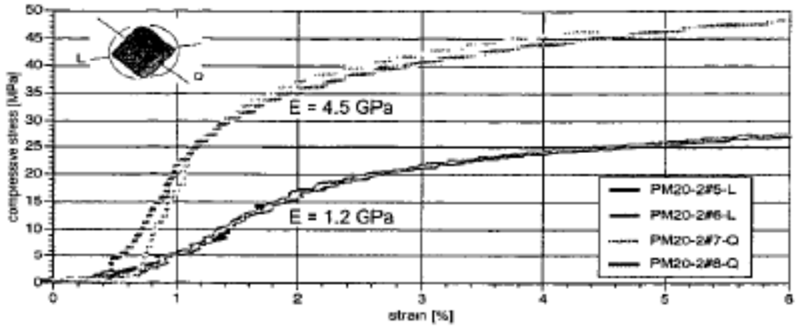
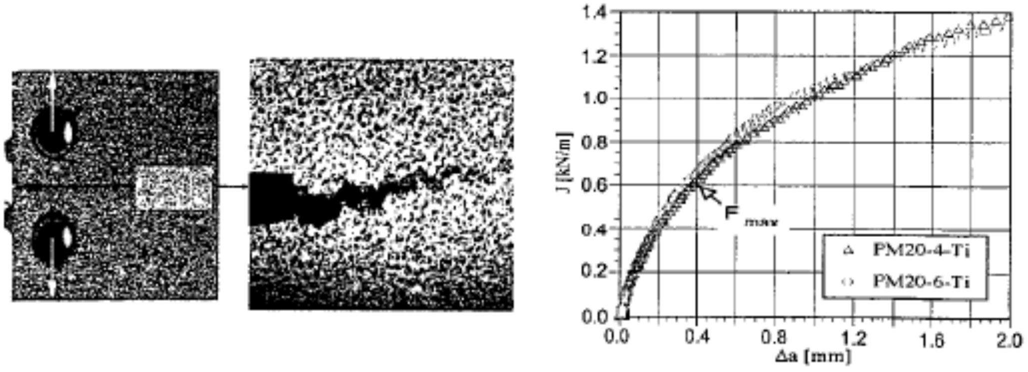


Fig. 6: Stress-strain curves for titanium samples (12x12x10 mm^3) with 68.4% porosity by compression along and across the compaction direction.

This diagram shows the effect of the deformation (loading) path as a higher strength and stiffness in the transversal direction. This can be explained by the morphology of the pores as shown in Fig. 4. The nearly elliptical shape of the pores provides rapid localisation and band development of the plastic strains in the narrow deformation area. This band spreads across the entire cross-section, leading to the early plastic collapse of the samples loaded in the L-direction. On the contrary, the transversally loaded samples exhibit discrete highly strained regions which do not tend to the formation of single or multiple bands. It appears that the formation of plastic strain bands causes significant reductions in yield strength and stiffness of porous material as shown in Fig. 6. This behaviour has a clear influence on the design of the loaded porous components. Here, there is a need to investigate the crack initiation and its growing with respect to the compaction direction of the materials. This work is still in progress.

Views of the fracture toughness samples and a fractured ligament of the porous titanium are shown in Fig. 7a. The fracture toughness was evaluated in the terms of J-integral determined using standard ASTM procedure. The J R-curves showing the resistance to ductile crack propagation are given in Fig.7b.



a.)

b.)

Fig.7a.) CT specimen and fractured ligament and **b.)** J R curves for two titanium samples.

The rising R-curve behaviour of the porous titanium can be of great interest for designing of the loaded components. The presence of the large amount of pores (some of them are connected to each other) in the basic material could cause a flat crack resistance curve as has been detected for some aluminium foam metals having much larger pores. Therefore, the rise of the R-curve can be explained due to the relatively small pore size and due to the crack tip "bridging effect" of the remotely deformed zones, which may contain microcracks ahead of the crack tip. Further investigations are still in progress to explain the governing micromechanism of the ductile fracture process at the crack tip for the highly porous titanium. Preliminary results have shown that the crack propagation mainly occurs after attainment of the maximum load, since crack growth of only about 0.4 mm was recorded at this point.

Conclusions

- Successful production of highly porous titanium parts by the powder metallurgy route is possible by using pore-forming additives. Special attention should be paid to the increase of their green density after compaction and pore-former removal. This can be achieved by special selection of the starting powders. Basic titanium powder should have an irregular particle shape. The size of its particles should be several times smaller than the size of pore-former particles. This contributes to the increase of the green strength and the number of contacts between particles of the basic material.

- The pore-forming additive used in this investigation acts as a pressing aid especially at high concentration. This peculiarity makes the technology easier, because there is no need to remove an additional pressing aid like wax. Furthermore, one of the impurity sources is excluded, which is particularly important in the production of titanium parts.

- The density increase by sintering porous titanium after removal of the pore-former is relatively small and corresponds to the shrinkage of Ti powder compacted without additive. This means that densification by sintering mainly occurs inside the titanium network. The macropores remain nearly unchanged. It was found that the radial shrinkage of the compacts during sintering is almost independent of the content of the pore-forming additive. This phenomenon allows the PM production of graded structures (e.g. highly porous top layer onto a dense substrate) without failure at the interface.

- The highly porous titanium demonstrates the anisotropy of the mechanical behaviour with respect to the compaction direction. The mechanical strength and elasticity modulus are sufficiently higher in the direction perpendicular to the compaction in comparison to these properties measured in the compaction direction.

- The fracture toughness investigation for porous titanium in terms of J-integral exhibits rising R-curve behaviour. This may be explained by the relatively small sizes of pores and/or "bridging effect" of the remotely deformed/cracked regions located ahead of the main crack tip.

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