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Novel porous Ti-Ta material with a micro core-shell structure using the powder metallurgy method

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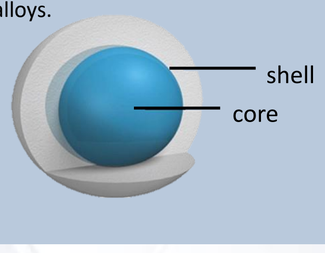
Core–shell structured materials have been extensively utilized to develop new functional materials and have become a research focus area in materials science. Pure titanium or tantalum has become one of the most favourable biomaterials for use in medical fields due to their much lower elastic moduli, excellent biocompatibility and superior corrosion resistance compared to more conventional used metallic biomaterials, such as stainless steels and cobalt based alloys.



AFTER MILLING

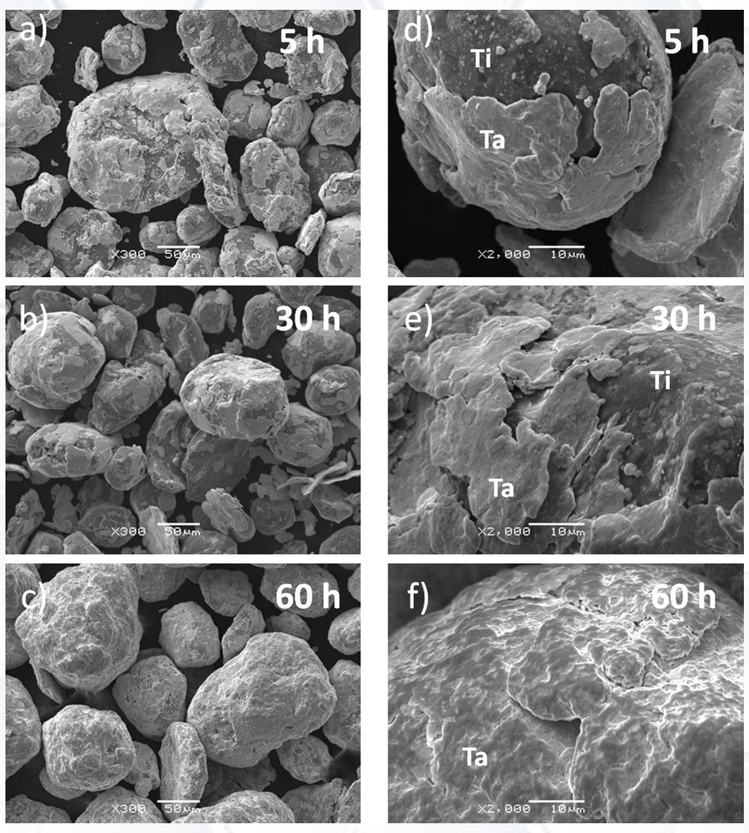
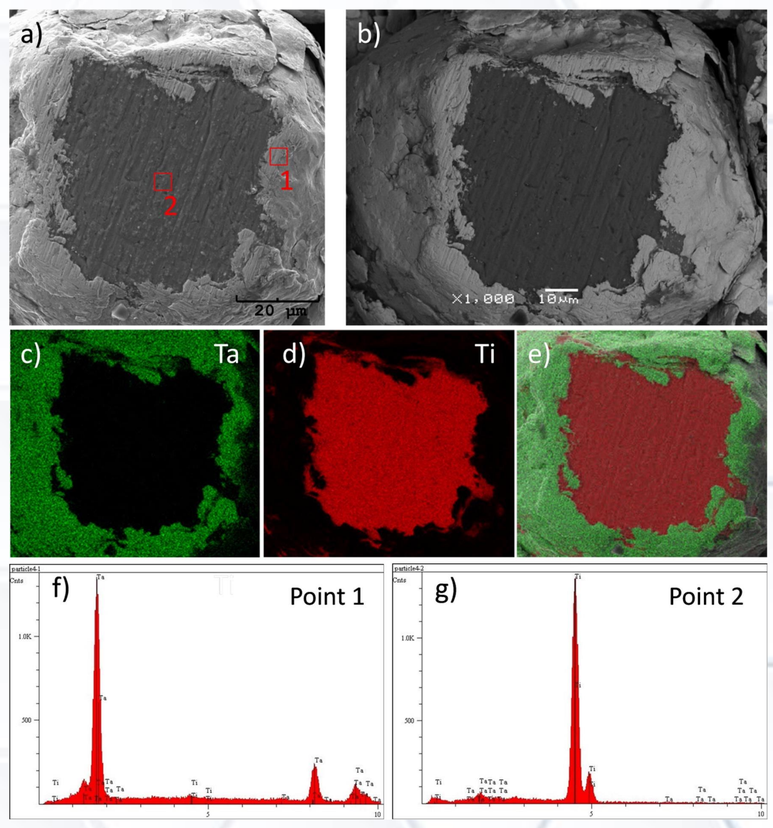


Fig 1. SEM images of the powders after: 5 (a and d), 30 (b and e) and 60 (c and f) hours of milling.



Fi. 2. Cross-sectional microphotograph of the core-shell structure of the as-prepared particle (a), backscattered electron SEM micrograph (b), distribution maps of the elements (c–e) and EDS spectra of points: 1 (f) and 2 (g).

The morphology of the core-shell powders after various milling times are presented. The microphotographs show progressive changes in the morphology of the tantalum shell on the titanium core for different milling times. During the first stage, a gradual flattening of the Ta particles is observed, and the particlesevolve to cover the titanium. At the further stage, the tantalum shell overlaps onto core particle, and eventually forms a uniformand fully covered core-shell powder. Intense plastic deformationand cold welding of the tantalum on titanium particle.

AFTER SINTERING

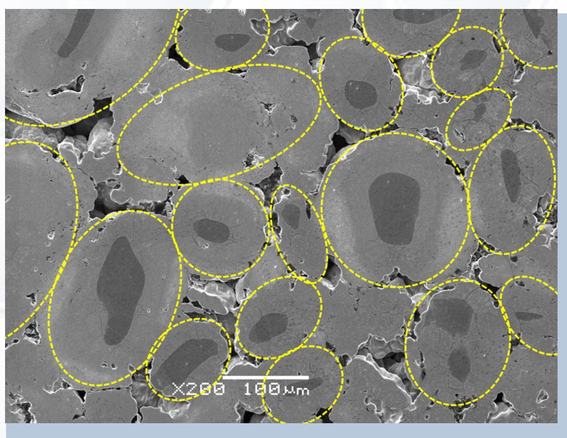


Fig. 3. SEM microphotographs with yellow marked particles in the sample after sintering

The porous microstructure of the material is characterized by spherical or elliptical shaped core-shell particles.

Pores were observed at the particle boundaries between the “connecting necks” of the particles. Additionally, smaller pores were observed inside the particles from the milling process. The metallographic cross-sections of the particles revealed the presence of various microstructure zones depending on the position inside the particles.

The particle consists of three areas including a dark core, a brighter transition area and a grey shell. The change of Ta content as a function of the core centre distance was analysed using EDS. The tantalum content increases with the distance from the core centre

The bright transition zone between the core and the outer shell exhibits a two-phase (α + β) Widmanstätten-type microstructure. The bright regions are Ta-rich (β-phase) and the dark regions are Ti-rich (α-phase). As the distance from the core increases, the thickness of the α phase plates decrease in favour of the β phase. Finally, a needle-like structure was revealed for the almost equilibrium zone.

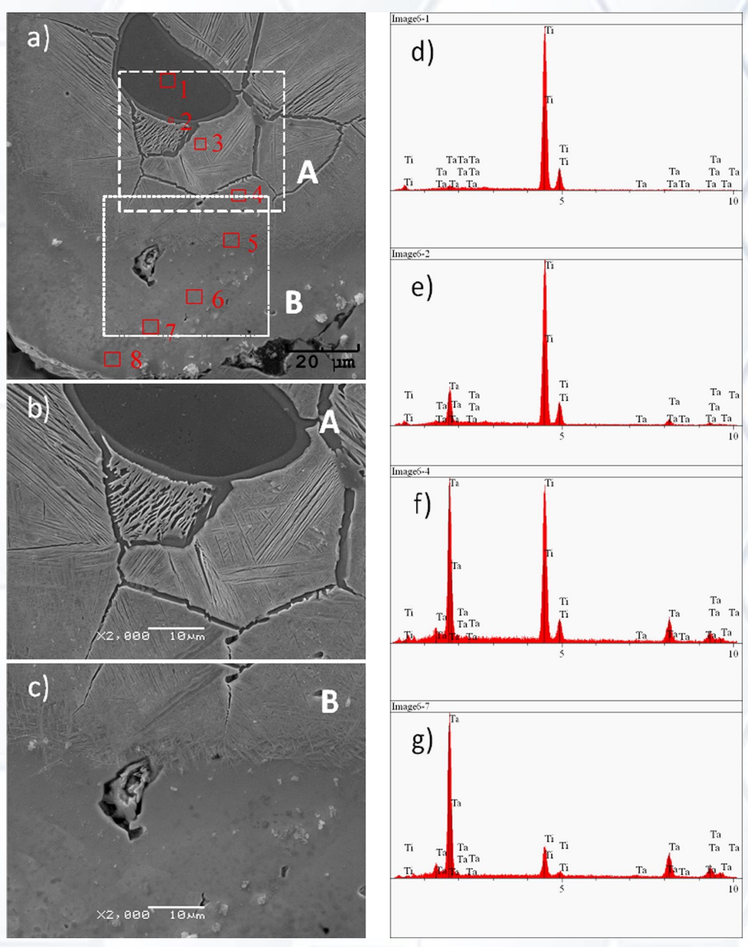


Fig 4. Enlarged SEM microphotographs of the particle cross-section (a), magnification of A (b), B (c) regions and EDS spectra of points: 1 (d), 2 (e), 4 (f) and 7 (g)

Fig 5. Topography and profile of the particle surface determined by AFM microscopy: 2d (a) and 3d (b).

MECHANICAL PROPERTIES

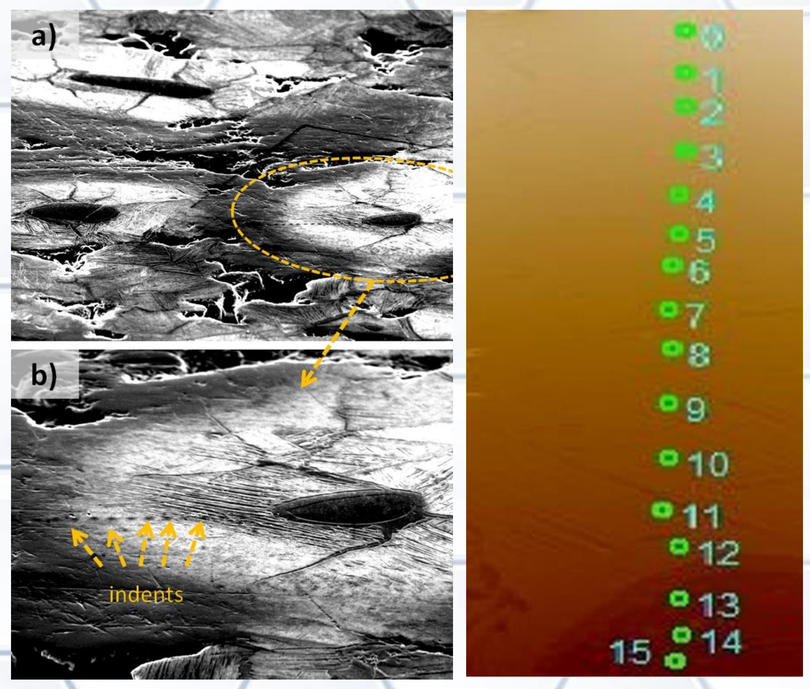


Fig 6. Enlarged SEM tilt microphotographs

Fig 7. The predefined points across the cross-section of the shell, transition area and core used for nanoindentation measurements.

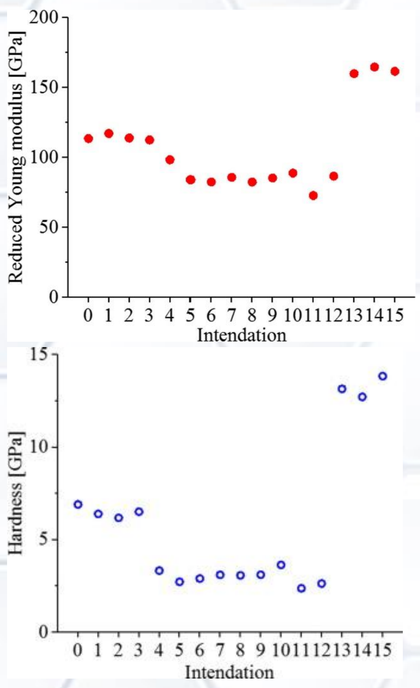


Fig 8. Dependence of the hardness and Young modulus on the indentation points cross the sample

It is worth mentioning that in this study, the hardness of the shell and core are higher than those for commercially used CP-Ti bio-material (2.55 GPa). Only the transition zone has similar hardness values as CP-Ti. The first areas include indents 0,1,2,3 corresponding to hardness values of approximately 7 GPa. The second area, (the transition area, indents 4–12) exhibited a hardness of approximately 3 GPa of hardness. It is worth noticing that the last two indents (those closest to core) revealed slightly lower hardness values corresponding to a different characteristic curve for 12 indent. The last area was the core (indents 13–15), which exhibited the highest hardness, reaching approximately 13 GPa, whereas the nanoarea hardness for pure Ti in the literature was approximately 4 GPa. The reduced Young modulus depends on the nanoindentation site. For the reduced Young's modulus, the core hardness exhibits a much higher value than the CP-Ti (107.33 GPa). The shell has similar values for the reduced Young's modulus, whereas the transition zone has a significantly lower value. The measurement of the reduced Young’s Modulus for first three indents revealed values between 110 GPa and 120 GPa. For further indents, the value of reduced Young’s Modulus were reduced to approximately 70 GPa, which is close to that of human bone.

STRUCTURE DETERMINATION

To investigate the structure of the core-shell particles in more detail, observations using transmission electron microscopy were carried out. Field “A” shows the transition area between the core and the shell. Fig. 10b shows two different areas: continuous and fine needle-like phases. The EDS analysis confirmed that the continuous phase was pure titanium. The TEM analysis of the transition zone revealed that the needle-like fine structure was α phase (bright phase) and the matrix was a near equilibrium of Ti and Ta (dark phase). Based on the TEM EDS result, we see that the tantalum contents for the “bright” and “dark” phases were 13.4(5) and 46.4(6) wt.%, respectively.

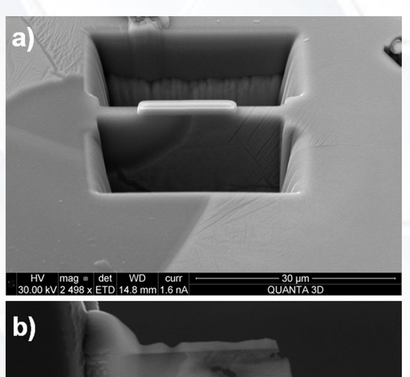


Fig 9. Cross-sectional views: (a) foil (b) and obtained by FIB

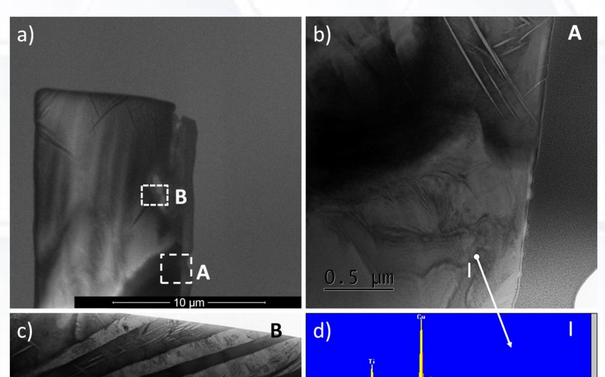


Fig 10. Micrograph of the foil (a) with marked places for the bright field TEM images (c and d) and EDS results (d–f).