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Effect of Annealing Temperature on Microstructure and Mechanical Properties of Hot Swaged cp-Ti Produced by Investment Casting

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The purpose of this study is to evaluate the influence of hot swaging (SW) and annealing treatment on microstructure and mechanical properties of commercially pure titanium (grade 4) produced by investment casting. During SW at 700 °C, the diameter of the cast titanium bars was reduced from 25 to 8.5 mm in 14 steps. After SW, material was annealed for 1 h at 500, 700, or 870 °C. The as-cast samples showed a typical microstructure consisting of a variety of α-morphologies, while the hot swaged samples exhibited a kinked lamellar microstructure. Annealing at 500 °C did not significantly change this microstructure, while annealing at both 700 and 870 °C led to recrystallization and formation of equiaxed microstructures. The cast bars exhibited a typical hard α-layer in near-surface regions with maximum depth and maximum hardness of 720 μm and 660 HV0.5, respectively. Due to SW, the tensile strength of the as-cast material drastically increased from 605 to 895 MPa. Annealing at 500 °C decreased this tensile strength slightly from 895 to 865 MPa while annealing at 700 °C led to a further pronounced drop in tensile strength from 865 to 710 MPa. No additional decrease in tensile strength was observed by increasing the annealing temperature from 700 to 870 °C. The tensile ductility of the as-cast and hot swaged samples was approximately the same in the range of 0.05 to 0.11, while the annealed samples showed values in the range of 0.25 to 0.53. In addition, the as-cast and hot swaged samples revealed a brittle cleavage fracture surfaces. However, the annealed samples showed a transgranular ductile fracture with formation of dimples.

Keywords investment casting, mechanical properties, pure titanium, swaging, thermomechanical processing

1. Introduction

Titanium alloys offer a unique combination of mechanical and physical properties as well as excellent corrosion resistance which make them desirable for a variety of critical applications (Ref 1-3). Elevated strength to weight ratio is the primary incentive for selection and design into aerospace applications including engine and airframe components. The expansion of titanium applications to non-aerospace industries, such as automotive, chemical, energy, marine, biomedicals, sports and architecture, entails improvements in the understanding of titanium metallurgy, advances in processing methods, ability to manufacture components without defects, and development of low-cost titanium alloys (Ref 4, 5). Commercially pure titanium (cp-Ti) is one of the most important titanium materials and widely used in many areas such as chemical, nuclear, and especially biomedical industries (Ref 6-8). The high cost of titanium makes net shape manufacturing routes very attractive.

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Casting is a near net shape manufacturing route that offers significant cost advantages over forgings or fabricated structures (Ref 9). The phase transformations that occur in titanium and its alloys make understanding of solidification and microstructure development more difficult than that of many other foundry alloys. Under equilibrium conditions, solidification in pure titanium occurs at 1668 °C. At 882 °C, this high temperature BCC β-phase transforms into the HCP low temperature α-phase (Ref 10). The microstructure of castings made of cp-Ti normally consists of equiaxed prior β grains, and some castings exhibit a small columnar zone at the mold wall. Within each equiaxed prior-β grain, a multitude of various α-morphologies exist; including Widmanstaetten, acicular, grain-boundary, lamellar, serrated, and plate-like α (Ref 11-14). Moreover, the as-cast cp-Ti microstructure yields little information about the solidification aspects of the high temperature β-phase. Only prior-β grains remain, and within these grains exists various α-morphologies. The prior-β grains remain visible because various α-morphologies nucleate from these boundaries, including the grain boundary α. In addition, the α-phase does not cross prior-β grain boundary (Ref 10).

Investment casting is considered one of the casting techniques that are used intensively for producing net shape components. Investment castings are applied in the automotive, aerospace, and biomedical industries for production of complex shapes (Ref 15-17). In this process, the shape to be produced is formed in wax and coated in a chemically bonded ceramic investment material. The investment is then heated to remove the wax. This process has the advantage of producing complex shapes at relatively low cost, scalability from single items to large numbers of products, and low wastage of raw materials (Ref 18). In titanium casting, pores are developed by gas entrapment and metal shrinkage upon solidification. Since volumetric shrinkage of titanium during solidification is high, approximately 3.5%, some of the total volumetric shrinkage will be converted to porosity (Ref 19) if casting procedures are not precisely controlled. This type of porosity is quantitatively predictable, concentrated in the areas that solidify last and strongly related to the geometry of the casting and design of feeding sprues (Ref 20).

This study was undertaken to cast cp-Ti in ceramic mold using investment casting technique. Hot swaging (SW) and annealing treatment were applied to enhance the mechanical properties of the cast samples. Microstructure and mechanical properties of the as-cast, hot swaged, and annealed conditions were studied.

2. Experimental Work

Commercially pure titanium (grade 4) samples were produced by melting in a vacuum induction skull melting furnace. This was done twice to get homogenous material. Investment casting technique was used to prepare two bars with diameters and lengths of 30 and 300 mm, respectively. An aluminum die was first prepared to form the wax pattern and then a ceramic mold was built on, as shown in Fig. 1(a). The ceramic mold was heated to 900 °C by means of a mold heater inside the induction furnace. The cast cp-Ti bars are shown in Fig. 1(b). In order to investigate the influence of ceramic mold on the structure of the cast cp-Ti alloy, the samples were longitudinally sectioned and photographed using stereo-microscopy. These bars were pre-conditioned by removing 2.5 mm from the surface through turning to get rid of the hard α-layer and to smooth the surface. As illustrated in Fig. 2, SW at 700 °C was applied to reduce the bars diameter from 25 to 8.5 mm. This was done using 14 steps with intermediate re-heating of 2 min. Part of the SW samples was annealed at 500, 700, and 870 °C for 1 h. The microstructures of the various conditions were investigated using optical microscopy. Tensile properties of the as-cast, SW, and SW + annealed conditions were evaluated. Three samples were tested and the average was taken. The fracture surfaces of the tensile samples were examined using scanning electron microscopy.

Fig. 1 Cast cp-Ti bars by investment casting. (a) Ceramic mold and (b) cast bars

3. Results and Discussion

The chemical composition of the cast bars is given in Table 1. All measured element concentrations are within typical values of cp-titanium (grade 4) reported in the literature (Ref 11). The as-investment cast microstructure is shown in Fig. 3(a). The microstructure consists mainly of equiaxed prior β grains and a variety of α-morphologies inside these grains. The α-morphologies at different locations can be identified as (A) grain boundary α, (B) fine acicular α, and (C) Widmanstaetten α (Fig. 3a). As shown in Fig. 3(b), the as-swaged microstructure is characterized by bent and kinked lamellae resulting from the severe plastic deformation applied during rotary swaging at 700 °C. Figure 4 illustrates the SW microstructures after annealing at temperatures ranging from 500 to 870 °C. Annealing at 500 °C (Fig. 4a), 700 °C (Fig. 4b), and 870 °C (Fig. 4c) leads to equiaxed grain sizes of roughly 12, 32, and 41 μm, respectively.

The cross-sectional morphology of the as-cast material is shown in Fig. 5. As expected, a reaction layer being caused by oxygen pick-up during casting is clearly visible in near-surface regions of the investment cast bars (Fig. 5a). Below this oxygen-enriched layer, the typical lamellar microstructure is formed (Fig. 5b). This gradient in microstructure from the surface to the interior is also reflected in the micro-hardness-depth distribution as shown in Fig. 6. The hardness decreases from a maximum of 660 HV0.5 at the outer surface gradually to a constant value of 265 HV0.5 as a distance of about 720 μm from the surface is reached. This hard oxygen-induced alpha case is unavoidable in investment casting of Ti-alloys and can also reach hundreds of microns in other titanium alloys (Ref 21).

The tensile properties of the as-cast, SW, and SW + annealed conditions are listed in Table 2. Swaging increases the ultimate tensile strength (UTS) of the as-cast condition by as much as 290 MPa from 605 to 895 MPa. This increase in strength is due to the swaging-induced high dislocation densities and the refined microstructure (compare Fig. 3, 4). Annealing at 500 °C for 1 h only slightly decreases this high-strength from 895 to 865 MPa while annealing at the higher temperatures of 700 and 870 °C showed a marked loss in strength amounting to 250 and 245 MPa, respectively (Table 2).

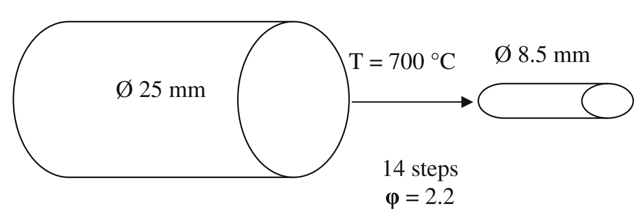

Fig. 2 Bar reduction by swaging (schematically)

Table 1 Chemical composition of the investigated cp-Ti (wt.%)

Al	Fe	Mo	Ni	Si	Sn	V	Ti
0.0014	0.088	0.004	0.008	0.0053	0.0126	0.002	99.9

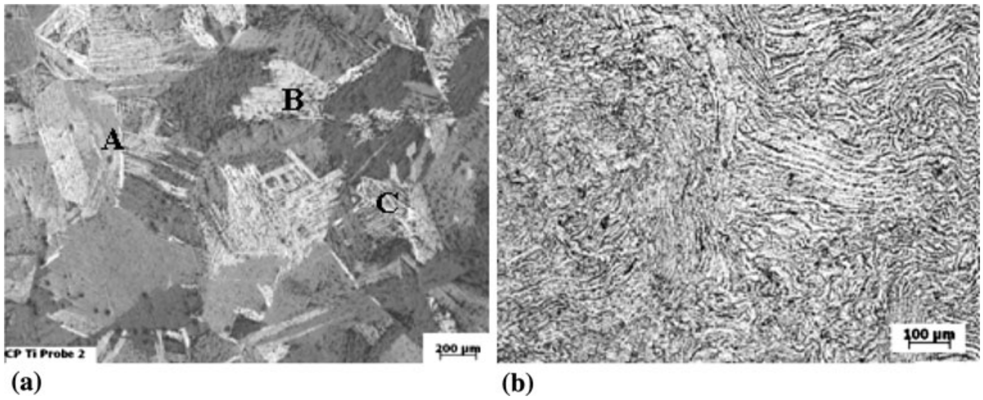

Fig. 3 Microstructures of cp-Ti. (a) As-investment cast and (b) as-swaged

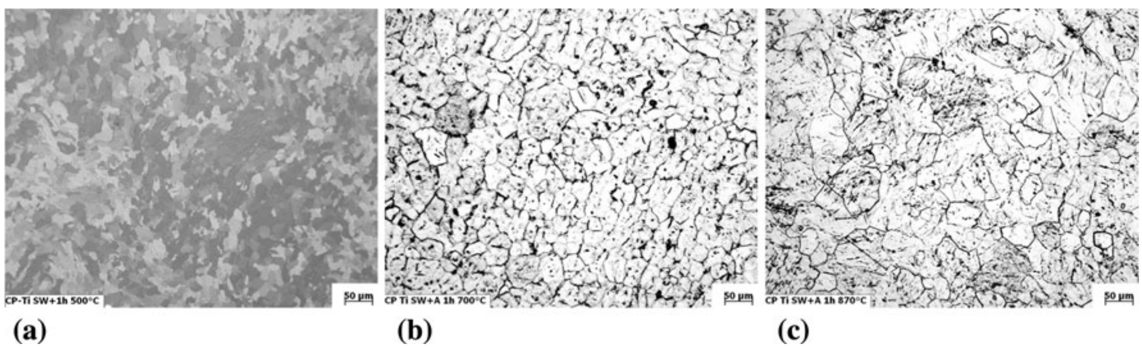

Fig. 4 Microstructures of the SW samples annealed at various temperatures for 1 h. (a) 500 °C, (b) 700 °C, and (c) 870 °C

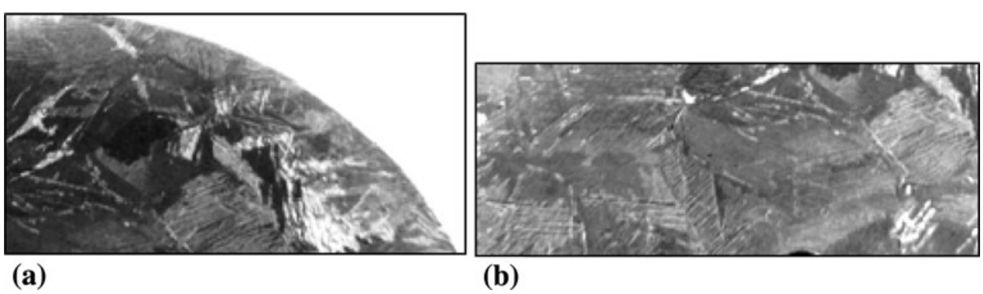

Fig. 5 Cross-sectional macrostructure of the as-cast bars. (a) Near-surface region and (b) central region

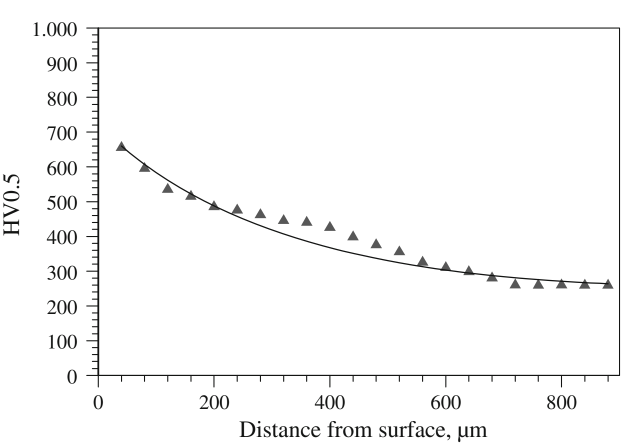

Fig. 6 Microhardness-depth profile in as-cast bars

Table 2 Tensile properties of the various conditions of cp-Ti

Condition	YS, MPa	UTS, MPa	$\varepsilon_F = \ln(A_o/A_F)$
As-cast	535	605	0.05
Hot swaged	815	895	0.11
Annealed at 500 °C/1 h	770	865	0.25
Annealed at 700 °C/1 h	555	715	0.53
Annealed at 870 °C/1 h	515	710	0.50

As expected, the tensile ductility of the as-cast condition is the lowest with $\varepsilon_F = 0.05$. After hot swaging, this value clearly increases to $\varepsilon_F = 0.12$ due to breaking down the as-cast coarse microstructure. The effect of the annealing temperature on YS, UTS and ε_F is illustrated in Fig. 7. YS as well as UTS gradually decrease with an increase in annealing temperature. As the strength values decrease, the tensile ductility markedly increases. With an increase in annealing temperature, the difference between UTS and YS clearly increases amounting to 195 MPa at the highest annealing temperature of 870 °C (Fig. 7). However, the difference between UTS and YS was estimated to be 80 MPa at ambient temperature (SW-condition).

The fracture surfaces of the tensile samples in the various conditions were carefully examined by SEM, as illustrated in Fig. 8(a) to (d). The as-cast samples showed typically cleavage fracture that normally occurs along a crystallographic plane, which in α titanium is the basal plane (Ref 22). Because the propensity for cleavage fracture decrease with decreasing grain size, the fracture surface of the SW samples showed cleavage facets with large amounts of dimples at the grain boundaries, Fig. 8(b). The annealed samples at 700 and 870 °C showed a transgranular ductile fracture with a strong formation of dimples due to the high ductility (Fig. 8c, d).

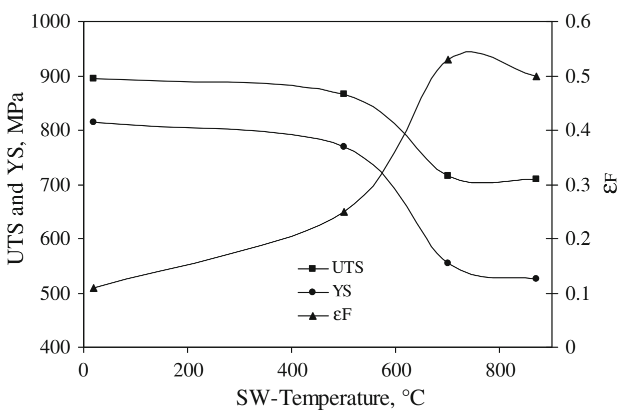

Fig. 7 Effect of annealing temperature after SW on the tensile properties

4. Summary

The microstructure of the as-cast cp-Ti contains a variety of α-morphologies; grain boundary α, fine acicular α, and Widmanstatten α inside the prior β-grains. The hot swaged samples showed a kinked lamellar microstructure with existing of α-phase flow due to the superplasticity during the swaging process.

The as-cast bars showed a hard α-layer on the surface of about 720 μm and the maximum hardness on the outer layer of the investment as-cast bars was 660 HV0.5, while base metal hardness was 265 HV20.

As-cast samples obtained a tensile strength of 605 MPa, which is markedly increased with swaging up to 895 MPa and further annealing up to 870 °C results in a loss of the strength to 710 MPa. The relative fracture ductility of the as-cast conditions is significantly increased with SW and SW + annealing.

As-cast and hot swaged tensile samples obtained a brittle cleavage fracture surface, while the annealed samples showed a transgranular ductile fracture with formation of dimples.

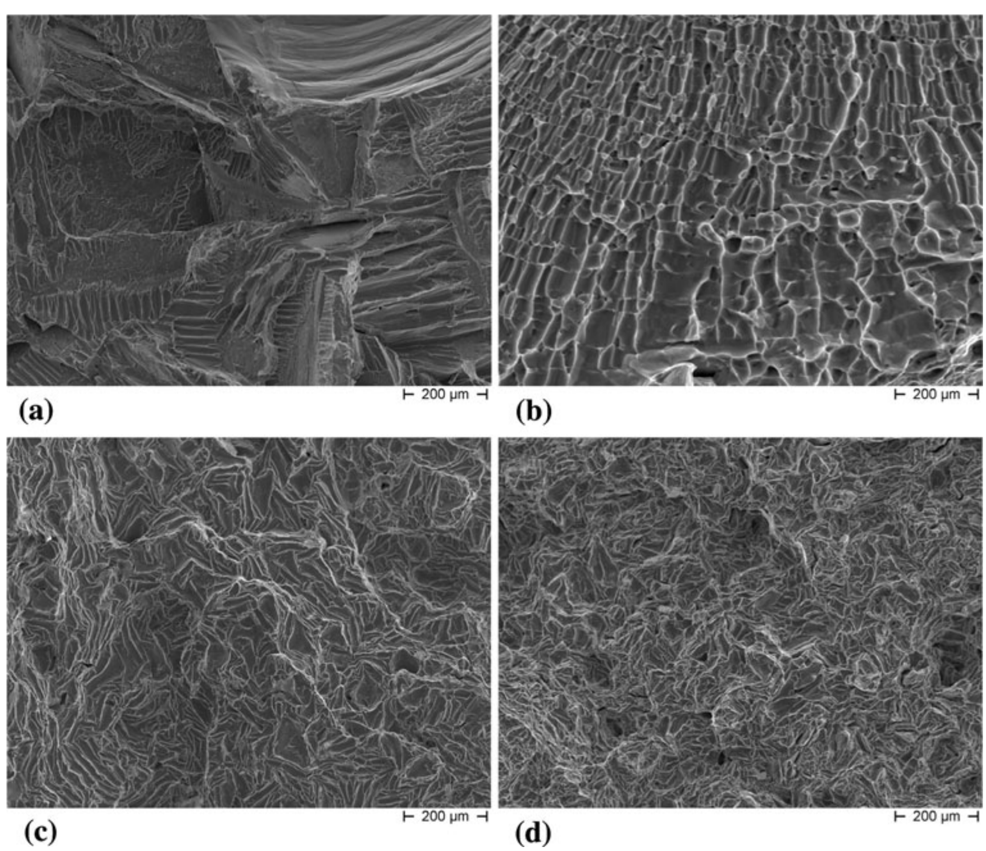

Fig. 8 Tensile fracture surfaces of cp-Ti. (a) As-cast, (b) SW, (c) SW + 700 °C, and (d) SW + 870 °C

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References

1. S. Tamirisakandala, B.V. Vedam, and R.B. Bhat, Recent Advances in the Deformation Processing of Titanium, *J. Mater. Eng. Perform.*, 2003, **12**, p 661–673
2. M. Balazic, J. Kopac, M. Jackson, and W. Ahmed, Review: Titanium and Titanium Alloys Applications in Medicine, *Int. J. Nano Biomater.*, 2007, **1**, p 3–34
3. W.W. Cheng, C.P. Ju, and J.H. Lin, Structure, Castability and Mechanical Properties of Commercially Pure and Alloyed Titanium Cast in Graphite Mould, *J. Oral Rehabil.*, 2007, **34**, p 528–540
4. N. Clement, A. Lenain, and P.J. Jacques, Mechanical Property Optimization Via Microstructural Control of New Metastable Beta Titanium Alloys, *JOM*, 2007, **59**, p 50–53
5. J.G. Ferrero, Candidate Materials for High Strength Fastener Applications in Both the Aerospace and Automotive Industries, *J. Mater. Eng. Perform.*, 2005, **14**, p 691–695
6. M.J. Tan and X.J. Zhu, Microstructure Evolution of cp Titanium During High Temperature Deformation, *J. Archiv. Mater. Sci. Eng.*, 2007, **28**, p 5–11
7. M.J. Tan and X.J. Zhu, Dynamic Recrystallization in Commercially Pure Titanium, *J. Achiv. Mater. Manuf. Eng.*, 2006, **18**, p 183–186
8. K. Watanabe, O. Miyakawa, Y. Takada, O. Okuno, and T. Okabe, Casting Behaviour of Titanium Alloys in a Centrifugal Casting Machine, *Biomaterials*, 2003, **24**, p 1737–1743
9. S.S. Rochn and G.L. Adabo, Vickers Hardness of Cast Commercially Pure Titanium and Ti-6Al-4V Alloy Submitted to Heat Treatment, *J. Braz. Dent.*, 2006, **17**, p 126–129
10. M.J. Bermingham, S.D. McDonald, M.S. Dargusch, and D.H. John, Microstructure of Cast Titanium Alloys, *Mater. Forum*, 2007, **31**, p 84–89
11. M.J. Bermingham, S.D. McDoland, M.S. Dargusch, and D.H. John, The Mechanism of Grain Refinement of Titanium by Silicon, *Scr. Mater.*, 2008, **58**, p 1050–1053
12. M.J. Bermingham, S.D. McDoland, D.H. John, and M.S. Dargusch, Beryllium as a Grain Refiner in Titanium Alloys, *J Alloys Compd*, 2009, **48**, p 120–123
13. M. Koike, Z. Cai, H. Fujii, M. Rezner, and T. Okabe, Corrosion Behaviour of Cast Titanium with Reduced Surface Reaction Layer Made by a Face-Coating Method, *Biomaterials*, 2003, **24**, p 4541–4549
14. M.J. Bermingham, S.D. McDoland, M.S. Dargusch, and D.H. John, Grain-Refinement Mechanisms in Titanium Alloys, *J. Mater. Res. Soc.*, 2008, **23**, p 97–104
15. R.C. Atwood, P.D. Lee, R.V. Curtis, and D.M. Maijer, Modeling the Investment Casting of a Titanium Crown, *Dent. Mater.*, 2007, **23**, p 60–70
16. D. Eliopoulos, S. Zinelis, and T. Papadopoulos, The Effect of Investment Material Type on the Contamination Zone and Mechanical Properties of Commercially Pure Titanium Castings, *J. Prosthet. Dent.*, 2005, **94**, p 539–548
17. H. Shimizu, T. Habu, Y. Takada, K. Watanabe, O. Okuno, and T. Okabe, Mold filling of Titanium Alloys in Two Different Wedge-Shaped Molds, *Biomaterials*, 2002, **23**, p 2275–2281
18. P. Oliveira, G.L. Adabo, R.F. Ribeiro, S. Rocha, F.A. Avila, and A.L. Valle, Influence of the Final Temperature of Investment Heating on the Tensile Strength and Vickers Hardness of cp-Ti and Ti-6Al-4V Alloy, *J. Appl. Oral Sci.*, 2007, **15**, p 44–48
19. M. Wu, J. Schadlich, M. Augthun, and H. Spielerman, Computer-Aided Prediction and Control of Shrinkage Porosity in Titanium Dental Casting, *Dent. Mater.*, 1998, **14**, p 321–328
20. D. Eliopoulos, S. Zinelis, and T. Papadopoulos, Porosity of cp-Ti Casting with Four Different Casting Machines, *J. Prosthet. Dent.*, 2004, **92**, p 377–381
21. C. Frueh, D.R. Poirier, and M.C. Maguire, The Effect of Silicon Containing Binders on Titanium/Face Coat Reaction, *Metall. Mater. Trans. B*, 1997, **28**, p 919–926
22. G. Luetering and J.C. Williams, *Titanium*, Springer, Berlin, Heidelberg, 2003