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The technology of precision casting of titanium alloys by centrifugal process

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Abstract

The article describes the development of a procedure for the preparation of foundry ceramic moulds and making first test castings.
The presented studies included:

- development of technological parameters of the ceramic mould preparation process using water-based zirconium binders and zirconia ceramic materials, where moulds are next used for the centrifugal casting of titanium alloys melted in vacuum furnaces,
- designing of pouring process using simulation software,
- making test castings,
- testing and control of the casting properties.

The technological process described in this paper enables making castings in titanium alloys weighing up to about 500 g and used in the majority of technical applications.

Keywords: Innovative Casting Materials, Titanium Alloys, Liquid Ceramic Slurries, Ceramic Mould

1. Introduction

So far, titanium alloys have been used as wrought materials. When titanium and its alloys are cast, very high requirements are imposed on moulding materials. The casting mould should be characterised by an exceptionally high thermal stability, total absence of any significant deformations and lack of chemical reactivity. Particularly undesirable is the tendency to the formation of layers of oxides and intermetallic compounds, deteriorating surface properties of castings.

To develop a technology of the ceramic mould preparation it has been decided to examine the applicability of:

- binders - colloidal sols of zirconium oxide ZrO_2, yttrium oxide Y_2O_3 and cerium oxide CeO_2,
- ceramic materials, i.e. flour and sand, and zirconia oxide stabilised with hafnium oxide.

2. Development of technological parameters for castings made from titanium alloys

The experimental work on the development of investment casting technology of Ti alloys required:

- testing of binders and liquid ceramic slurries,
- studies of technological properties of ceramic moulds,
- studies of pouring process using simulation software and of the pilot castings manufacture,
- control checking of obtained castings.

Physical and chemical characteristics of binders and of the liquid ceramic slurries included:

- rheological properties,
- determination of the characteristic phase transformation temperatures (i.e. the temperatures of sintering, softening, melting and flow).

The study of technological properties of the ceramic moulds included:

- determination of mechanical strength,
- measurements of surface roughness,
- high-temperature testing of the specific effect of selected ceramic moulding materials in contact with liquid titanium.

Making of castings was preceded by numerous computer simulations of the mould pouring process and metal solidification to develop an optimum geometry of the gating system.

Quality control of castings included:

- testing of mechanical strength,
- examinations of casting microstructure,
- examinations of casting structure by computed tomography.

3. Results

3.1. Measurements of thermophysical parameters of ceramic slurries

For the determination of characteristic temperatures of samples of the ceramic slurry, a PR-25/1750 apparatus called high-temperature microscope was used. This apparatus is applied in determination of the temperature of phase transformations (sintering, softening, melting and flow), which occur in ceramic materials in the temperature range of 800÷1750°C.

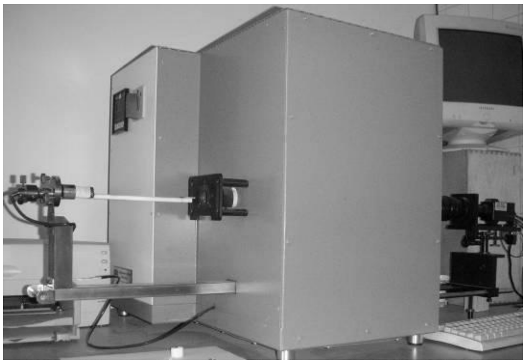


Fig.1. PR-25/1750 apparatus for the determination of characteristic temperatures

For analysis of the test results, a computer software, assisting the determination of the temperature of phase transformations, was used. Within the temperature range of 600 do 850°C, the heating rate was 15°C/min. When the temperature of 850°C was reached, the heating rate was reduced to 10°C/min. The recording and measurement of the characteristics were performed within the temperature range from 900 to about 1750°C; within the temperature range of 1650 to 1750°C, the heating rate was reduced to 8°C/min.

Samples for testing the temperature characteristics of ceramic materials were prepared in the form of 5 mm dia x 5 mm height cylinders, compacting the ceramic material in a special die for sample preparation (Fig. 2).

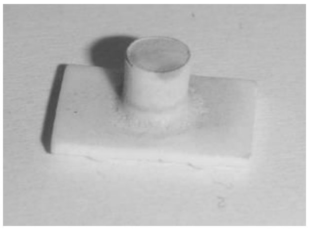
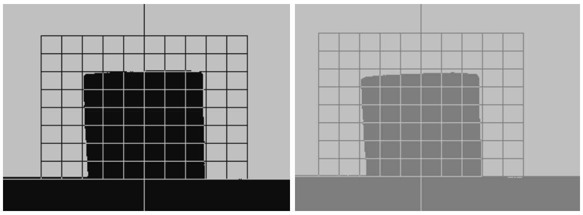


Fig. 2. Example of sample after baking

An example of the test results obtained for a sample of ZrO_2 is shown in Figs. 3 and 4.



a) b)

Fig. 3. An image of ZrO_2 sample: A) – at 903°C, B) - at 1750°C

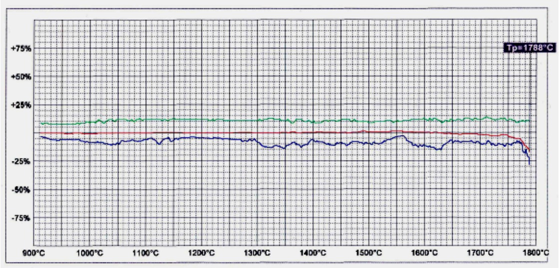


Fig. 4. The run of heating process and thermophysical parameters obtained for a sample of zirconium oxide ZrO_2

A sample prepared from ZrO_2 was characterised by favourable thermophysical parameters. A small change in the sample height (red line) occurred only when the temperature of 1650°C has been exceeded. Comparing the specimen shape at the beginning and end of the measurement shows very small changes only.

3.2. Testing and evaluation of the strength properties of the ceramic mould layers

The strength of ceramic mould layers is a characteristic feature inherently related with the grain size, grain size distribution, the sample size and shape, the method of sample preparation, and the research technique associated with the methodology of taking the measurements. Therefore, the developed method of the strength measurement takes into account the specific character of the preparation of the ceramic mould layers and enables determination of a correlation between the final mould strength and technological parameters of its preparation. As an output parameter in the performed tests, the type of binder and the specimen baking temperature were adopted.

The starting content of zirconia flour in total volume of the ceramic slurry was 45÷48%. The liquid ceramic slurry was homogenised for a period of 24 hours, and then within the time of approximately 48 hours, samples of the ceramic material used for the individual mould layers were taken. Samples of the ceramic slurry were prepared in accordance with the investment casting technology using wax moulds, designed in a way such as to make four ceramic samples simultaneously (Fig. 5). The ready liquid slurry was poured into moulds, and then after removing its excess the surface was sprinkled with ZrO_2 sand. The first and the second mould layer was made from ZrO_2 sand of the grain size comprised in a range of 0,1-0,3 mm. The next layers were made from the sand of the grain size 0,5÷1mm. After application of each layer, samples were dried. The samples were ready after application of 5 or 6 layers.

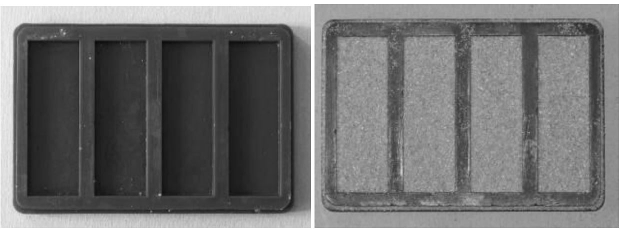


Fig. 5. Wax mould and samples of the layered shell moulds

After melting the wax at a temperature of 200 °C in a drier with forced-air circulation, raw samples of dimensions: length 50 mm, width 20 mm and height of 7 mm were obtained. The ready samples were used in:

- determination of surface roughness;
- determination of the ceramic slurry strength in function of temperature;
- microstructural examinations (SEM) and local determination of chemical composition in microregions by the method of X-ray microanalysis (EDS).

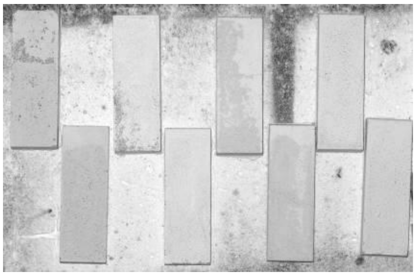
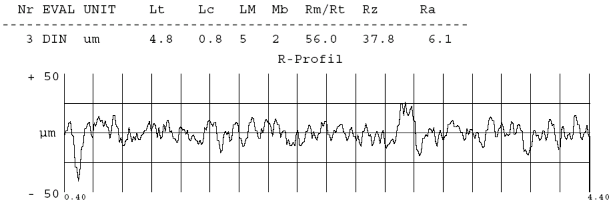


Fig. 6. Samples of ceramic mould layers

3.3. Surface roughness measurement

Surface roughness was measured with Hommel Tester T500 apparatus on layered ceramic samples baked at 1250°C. In accordance with the previously established methodology, the measurement was taken on the sample surface reproducing the surface of a wax mould. An example of the surface roughness profile for sample containing Y_2O_3 is shown in Fig. 8, while summary of the results is given in Table 1.



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Nr EVAL UNIT    Lt     Lc   LM  Mb  Rm/Rt  Rz      Ra
-------------------------------------------------------------
 3 DIN  um      4.8    0.8  5   2   56.0   37.8     6.1
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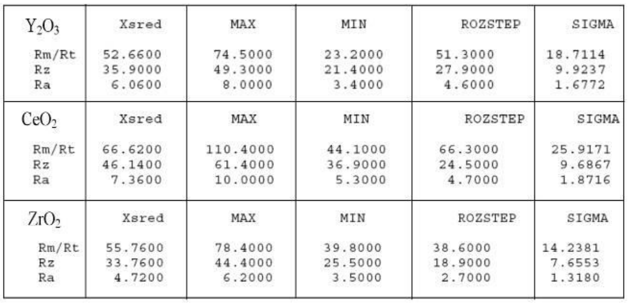
Fig. 7. Surface profile for sample with Y_2O_3 binder

Table 1.
Comparison of the results of surface roughness parameters measurement taken on samples of the layered ceramic materials



Y_2O_3	Xsred	MAX	MIN	ROZSTEP	SIGMA
Rm/Rt	52.6600	74.5000	23.2000	51.3000	18.7114
Rz	35.9000	49.3000	21.4000	27.9000	9.9237
Ra	6.0600	8.0000	3.4000	4.6000	1.6772
CeO_2	Xsred	MAX	MIN	ROZSTEP	SIGMA
Rm/Rt	66.6200	110.4000	44.1000	66.3000	25.9171
Rz	46.1400	61.4000	36.9000	24.5000	9.6867
Ra	7.3600	10.0000	5.3000	4.7000	1.8716
ZrO_2	Xsred	MAX	MIN	ROZSTEP	SIGMA
Rm/Rt	55.7600	78.4000	39.8000	38.6000	14.2381
Rz	33.7600	44.4000	25.5000	18.9000	7.6553
Ra	4.7200	6.2000	3.5000	2.7000	1.3180

where:
Ra – the arithmetic average roughness value (DIN4777);
Rz – the average roughness height;
Rmax – the max. roughness height;
Rt – the total height of the roughness profile,
Lt – the measuring length (4,8 mm)

3.4. High-temperature strength testing of ceramic mould layers

The strength of the ready samples of the ceramic materials was measured with an apparatus of original design developed by the Foundry Research Institute in Cracow.

To test the properties of ceramic mould layers, a four-point bending test method has been developed. The method of four-point bending enables loading a larger sample volume than the three-point test. With uniform load between supports it is easier to find critical defects, which can casually occur in the sample. The method of four-point bending test is considered more representative of the weakest link in the structure of a ceramic mould. In all these systems, the strength of the ceramic material is determined by the following formula (1):

$$\sigma_f = \frac{3}{2}\frac{F_f (L - l)}{d \cdot h^2} \qquad (1)$$

where:
σ_f - the sample strength (breaking stress on bending) [MPa];

F_f - the breaking load [N];
L - the distance between the supports [mm];
l - the load spacing [mm];
d - the specimen width [mm];
h - the specimen height [mm];

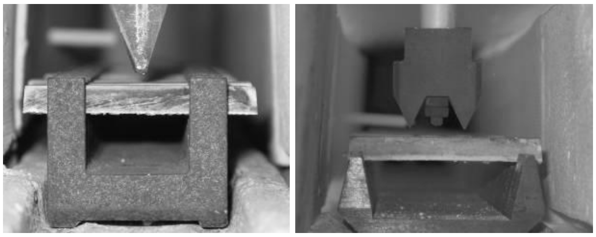


Fig. 8. Three- and four-point specimen loading systems

The results of measurements of the bending strength of samples of the ceramic slurry prepared with the examined binders are shown in Figure 9.

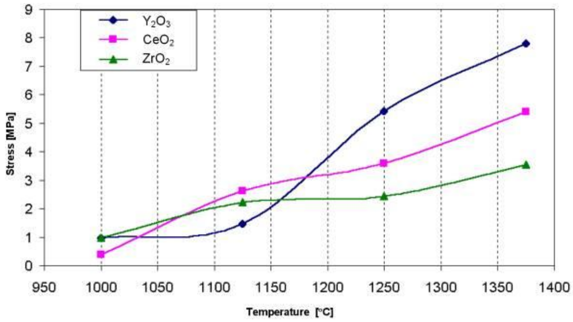
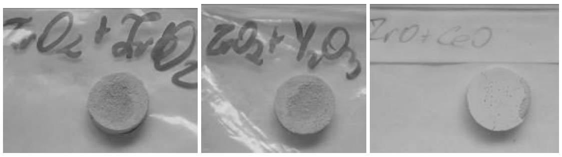


Fig. 9. The bending strength curve plotted for the ceramic slurry samples with different binders

3.5. Ceramic mould material interaction in contact with molten titanium alloy

High-temperature studies of the specific interaction of selected ceramic mould materials in contact with liquid titanium were carried out at the Centre for High Temperature Studies of Metals and Alloys operating in the Foundry Research Institute in Cracow. The sessile drop method was applied in a modified version of the contact heating of the examined materials couple, i.e. the samples of titanium and of the mould material substrate. This method consists in determining the kinetics of wetting the solid substrate by the tested liquid metal and involves recording of the drop image and measuring next the contact angle (θ) between the liquid and selected solid substrate.



a) b) c)

Fig. 10. Examined substrates:
a) binder - ZrO_2, ceramic material - ZrO_2;
b) binder - Y_2O_3, ceramic material - ZrO_2;
c) binder - CeO_2, ceramic material - ZrO_2;

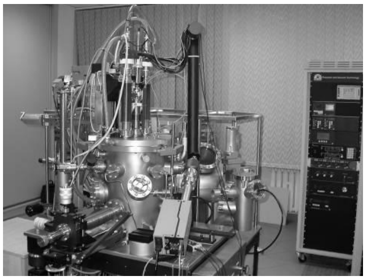


Fig. 11. A versatile set of apparatus for complex high-temperature studies of liquid metals and alloys

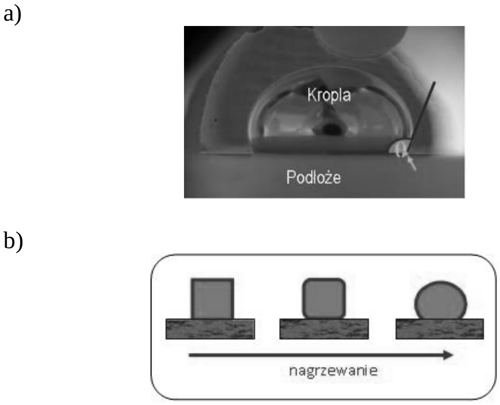
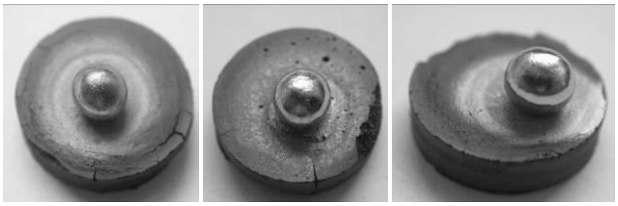


Fig. 12. Procedure of contact heating (CH) of the examined materials couple, a) example image of the examined object as recorded with digital camera; b) schematic diagram.



a) b) c)

Fig. 13. Photographs of samples after Ti wettability testing:
a) substrate – binder ZrO_2+ZrO_2, 1810°C, 2min;
b) substrate – binder CeO_2+ZrO_2, 1800°C, 5min;
c) substrate – binder Y_2O_3+ZrO_2, 1800°C, 5min

3.6. Computer-aided simulation of mould pouring and solidification process

The possibility of using centrifugal force in the process of filling foundry mould with metal can significantly improve the accuracy of the castings made. The centripetal acceleration value of a = 40 g is obtained on the circumference of a circle of 1 m diameter rotating at a speed of 270 rpm. In the case of castings made in centrifugal Titancast 700 and SuperCast furnaces, the length of ceramic mould is similar to the distance of the gating system from the axis of revolution. In this case, the process of mould filling with metal is determined by effects associated with the Coriolis force.

Problems related with the calculation of fluid flow in a spinning mould can be solved with a Flow3D software, having a rich database on the field of forces generated in a variety of the physical phenomena. As in every other numerical programme, the basic problem is correct selection of proper boundary conditions for the computations.

The application of FAVOR™ software enables dividing the computed spaces into a gradually thickening rectangular grid. The only thing to be remembered is to use integral multiples in changes of the scale.

Correct preparation of simulation requires initial analysis of parameters related with the casting process. Of course, the basis for such analysis is the previously designed geometry mapping the configuration of mould, gating system and pattern. In Flow3D software it is necessary to prepare the, so called, workspace, in which all the files related with the model geometry and description of boundary conditions will be placed.

Using "fit to geometry" option allows adjusting the grid to the model geometry. The number of grid elements should be selected in a way such as to enable exact model mapping during computations.

Having analysed the accuracy of the selected grid, it is necessary to determine next a mathematical model of the phenomenon, which the computational algorithm will use in simulation. Flow3D software is equipped with numerous physical models that allow simulation of various phenomena which occur when metal is poured into mould and solidifies.

The computations enable visual representation of the liquid metal flow in mould. An independent analysis of the velocity, pressure and potential structural defects is also possible.

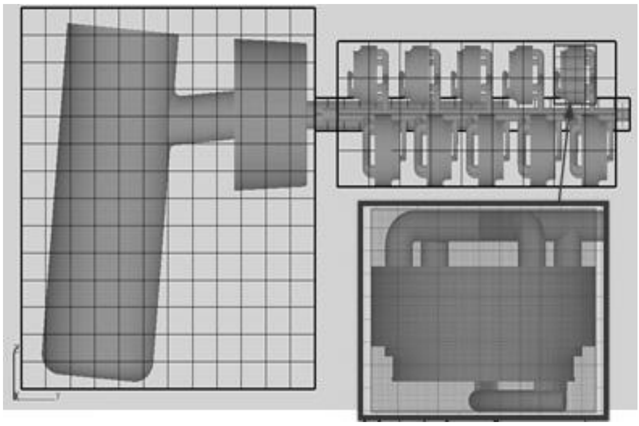


Fig. 14. The division of the examined volume into fragments of increasing accuracy of computations

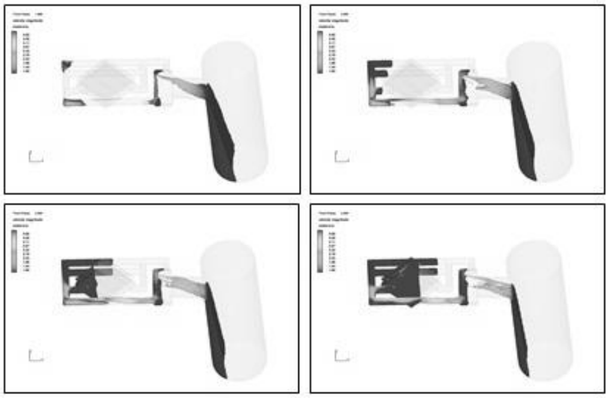


Fig. 15. Selected fragments of computations of the metal flow rate during centrifugal mould pouring

Multitasking of Flow3D software allows its application in the analysis of numerous processes taking place during casting, such as:

- gas evolution in the full mould process,
- porosity formation,
- solidification / melting,
- segregation,
- cyclic heating during casting in permanent moulds,
- cavitation,
- injection process in casting at elevated pressures.

The obtained results of computations enabled establishing the technique of filling the mould with liquid metal with foundry patterns of various geometries. A verification of the adopted solution was possible at the stage of designing a pattern set, which significantly facilitated the development of foundry technology.

3.7. Making test castings

3.7.1. Wax patterns

The investment casting process starts with the preparation of foundry pattern exactly reproducing the shape of the designed product.

The pattern material should be characterised by high dimensional stability and reduced to minimum reactivity with the applied ceramic coating. Important is also the low ash content. For small products with high dimensional tolerances it is preferable to use a jeweller's wax Castingaldo Jewelry Injection Wax.

When making a pattern set from the pattern mixture one has to remember that total volume of the set should be slightly larger than the molten metal volume. It is also necessary to take into account the technique of mould filling with metal, including the vent channels which fill as the last ones.

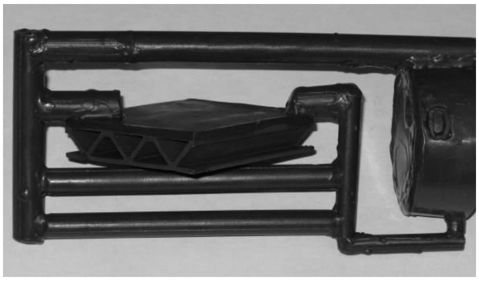


Fig. 16. Foundry pattern set

All the prepared sets were provided with accessories which enabled making specimens for mechanical testing.

The design of the manufactured pattern set was based on the results of earlier experience related with the centrifugal technique of mould pouring.

With pattern-making operations completed, each pattern was weighed and after careful degreasing was coated with a surface active agent to facilitate wetting of the wax surface with the water-based suspension of ceramic slurry.

3.7.2. Ceramic moulds

The liquid ceramic slurry prepared for the first mould layer was characterised by an outflow time of 50 s (Ford cup of 4 mm). The first layer was sprinkled with the sand of 0,1÷0,3 mm granulation. Then, the liquid ceramic slurry was diluted to obtain the outflow time of 25 s.

The results are shown in Figure 17.

Relevant volume content of flour in the liquid ceramic slurry is one of the factors that determine the ceramic mould life.

The liquid ceramic slurries with an addition of binders based on zirconia, yttria and ceria acquire their optimum viscosity at 47 vol. % of the flour content in solution.

The next mould layers were made from the sand of 0,5÷1mm grain size. Very helpful in the application of subsequent mould layers was the possibility to use a tumbling barrel for sand sprinkling.

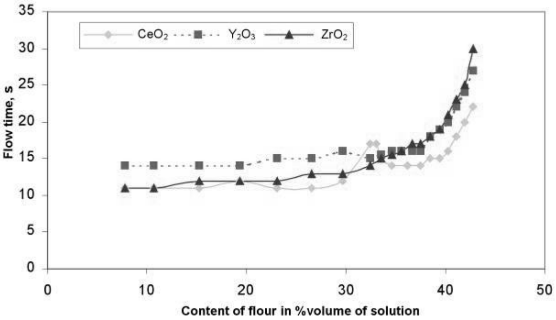


Fig. 17. Outflow time vs volume content of ceramic flour in solution

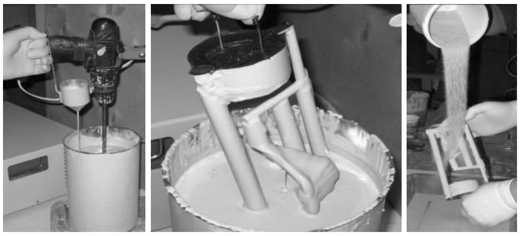


Fig. 18. Viscosity measurement with Ford cup. Making first layer of ceramic mould

Fig. 19. Making subsequent layers of ceramic mould

After the application of 10 layers of the ceramic slurry, the process was completed and the unnecessary top part of the ceramic mould was cut off. The pattern material was melted out in an autoclave in the atmosphere of water vapour at 130°C. The process of melting was running smoothly and no cracks, even the least ones, were observed in the ceramic moulds.

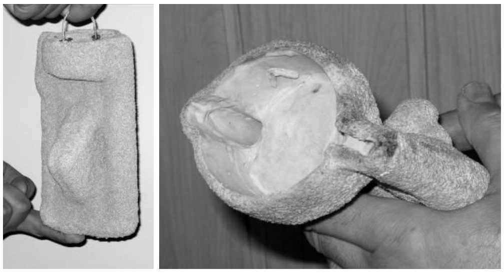


Fig. 20. Ceramic mould in layers. Mould after melting out of pattern material

Considering strong centrifugal forces acting during centrifugal pouring of moulds, the ready moulds were placed in a ceramic block mould made by the Shaw process. The outer sleeve made of perforated stainless steel enabled baking of moulds at 1250°C.

Fig. 21. The subsequent stages of foundry mould preparation

3.7.3. Making castings from TiAl6V4 alloy

Considering strong centrifugal forces operating during centrifugal mould pouring, the idea of making castings in self-supported moulds was abandoned. Moulds were placed in sleeves made of perforated stainless steel and filled with Shaw mixture. After initial drying, moulds were baked at 1000°C and then cooled to the pouring temperature. The ready charge of metallic TiAl6V4 was placed in crucible and carefully dried at a temperature of 200°C.

Fig. 22. Successive stages of melt preparation

The process of charge melting and mould pouring was performed in a Supercast centrifugal vacuum induction furnace from Linn High Therm allowing melting of up to 1,2 kg of titanium alloy.

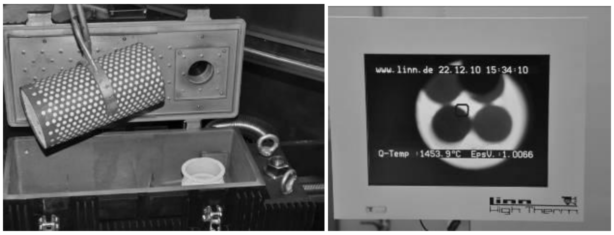


Fig. 23. Successive stages of the casting process. Charge loaded to the furnace chamber and titanium melting process monitoring

In furnace chamber, the next preheated crucible with metallic charge was placed, and then the hot metal sleeve with a ceramic mould. After closing the furnace chamber and rinsing it with pure argon, the process of charge melting started. Monitoring and control of the whole process were much easier looking at the picture obtained from a CCD camera connected to an optical pyrometer. When pouring temperature was reached, the generator coil was lowered and spinning of the chamber started to allow molten metal flow from the crucible into a mould. After completion of the spinning process, the furnace chamber was filled with liquid argon until the value of atmospheric pressure. It was possible to safely open the chamber and remove the metal sleeve poured with titanium alloy. The mould was left to cool down under ambient conditions, and then the ceramic shell was removed as well as the material forming mould itself.

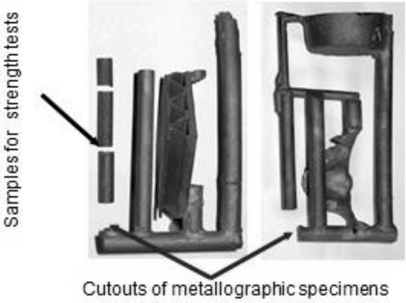


Fig. 24. Test castings ready – note the marked places where samples were taken for quality inspection

The individual elements of a foundry set were cut off with a cooled diamond saw and cleaned using a sand-blasting device and alundum sand of the 0,1 to 0,2 mm granulation.

3.8. Casting quality testing

3.8.1. Tensile strength

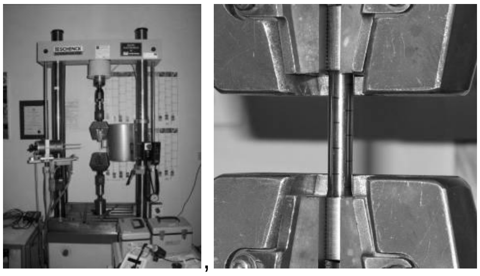


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Fig. 25. Tensile testing of cast Ti alloy specimens

The tensile strength of ϕ 10,5 mm rod cut out from the first casting exceeded 800 MPa. Considering the quality of surface layer and the natural porosity along the axis of the rod, the obtained result seems quite good.

3.8.2. Microstructural examinations

The following photographs show structure obtained on the specimen fracture and the results of metallographic examinations. The surface layer of 0.3 mm thickness is typical for titanium alloy castings. The thickness of the surface layer, conventionally called α-case, depends on the type of the used ceramics and on the metal cooling rate (thickness of the casting).

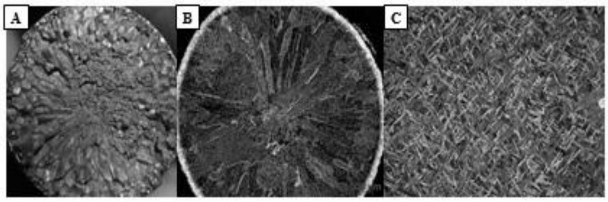

Fig. 26. Photograph of the fracture (A). Specimen microstructure (12.5x) (B). Specimen microstructure (100x) (C)

3.8.3. Structural examinations by computed tomography

Typical research techniques (determination of tensile strength and crystallography of microstructure) were completed with porosity measurements made by Nanotom 180 X-ray computer tomograph. The possibility to determine the homogeneity of the cast structure is of utmost importance, especially in the case of products for medical applications.

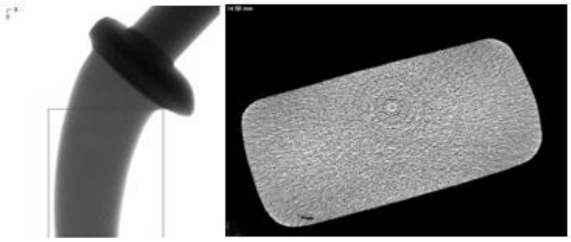

Fig. 27. Examination of cast structure by computed tomography

The result of the examination is a virtual spatial image of the scanned object, showing the exact location of both porosity and structure defects

4. Summary

The presented technological process enables making titanium alloy castings for the majority of technical applications. In the course of study, a technique of making investment pattern sets was developed, followed by a methodology of making the layered ceramic moulds for casting of titanium alloys. The technique of metal melting and mould pouring in a vacuum induction furnace for the centrifugal casting was specified.

New ceramic materials prepared with nanoparticles of Ce, Y, Zr oxides acting as binders showed the required chemical stability in contact with the liquid Ti. They can be used as a ceramic mould material for casting of Ti alloys.

The basic assumption was that the ceramic moulds would be made from liquid ceramic slurries containing zirconium binder, ZrO_2 flour and coarse zirconia. A technique for melting titanium alloy in a crucible made of zirconium material was developed. This melting technique allowed obtaining castings free from the defective surface layer containing carbon compounds.

Acknowledgments

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