Ulrich E. Klotz*, Tiziana Heiss and Dario Tiberto
fem - Research Institute for Precious Metals & Metals Chemistry

Dr. Ulrich E. Klotz is Head of the Department of Physical Metallurgy at the Research Institute for Precious Metals & Metals Chemistry (fem) in Schwaebisch Gmuend, Germany. He is a Diploma Engineer in Physical Metallurgy (University of Stuttgart, Germany) and holds a PhD in Materials Science from ETH Zurich, Switzerland.

B. Sc. Tiziana Drago has a Bachelor in Materials Engineering with the specialization in surfaces treatments (Politecnico di Milano, Italy) and a Master in Materials and diagnostic techniques in heritage manufactures (University of Pisa, Italy). She works in the Department of Physical Metallurgy at the Research Institute for Precious Metals & Metals Chemistry (fem) in Schwaebisch Gmuend, Germany.

Dario Tiberto has a Bachelor of Science in Mechanical Engineering (Politecnico di Torino). He works in the Department of Physical Metallurgy at the Research Institute for Precious Metals & Metals Chemistry (fem) in Schwaebisch Gmuend, Germany.

.

This paper summarises the results of a collaborative research work on investment casting of widely used platinum alloys (Pt-5Ru and Pt-5Co) for jewellery purposes. In order to allow the simulation of the casting process, a materials database was developed in a first step. Casting simulation tools based on computational fluid dynamics have been used for the optimization and deeper understanding of the role of the casting process parameters.
Selected casting trials with detailed monitoring of the casting process were conducted using industrial tilt and centrifugal casting machines. Dedicated tree setups for the different machines were optimized using the casting simulation tools. The form filling, the surface quality and the microstructure and porosity of the cast items were analysed to investigate the role of different casting parameters and geometrical conditions in the different casting setups. The casting simulation results were employed for a deeper understanding of the experimental casting results.

Platinum investment casting: material properties, casting simulation and optimum process parameters

Ulrich E. Klotz*, Tiziana Heiss and Dario Tiberto

Research Institute Precious Metals & Metals Chemistry (fem)

INTRODUCTION

In previous years the investment casting of platinum alloys has been empirically investigated. The aim of these investigations was a better understanding of the casting process and its optimization. The studies vary in terms of the applied casting methods, the alloy compositions and the tree setup used (1, 2, 3, 4). However, due to the high material costs and the limited distribution of platinum jewellery fewer studies compared to gold and silver alloys have been conducted. The simulation of the casting process by fluid dynamics (in this paper referred to as "casting simulation") proved to be a valuable tool in gold and silver investment casting [7, 8]. Casting simulation proved to be beneficial as a complementary tool to conventional empirical casting studies. The combination of casting simulation, casting experiments and characterization of the resulting cast parts results in a better understanding of the casting process and hence a better (quality) control. So far, the casting simulation of platinum alloys has not been published in open literature, although it is expected to have a huge potential for the optimization of process parameters and hence the reduction of costs. Systematic correlations between the process parameters and casting quality have not yet been quantitatively described. Some general knowledge can be adapted from gold and silver casting, but some largely different material parameters have to be taken into account. The aim of this work is to confer a remedy on this deficit. Specifically, it strives to understand und to optimize the influences of process parameters on the quality of Pt-5Ru and Pt-5Co casts with the aid of casting simulation. This paper records the results of a collaborative research project, supported by the German Federal Ministry of Economics and Technology (project no. AiF–IGF 16413N). The scope of this project was to develop a materials database enabling the casting simulation (form filling and solidification) of platinum alloys. To validate the simulation results, selected casting trials including detailed process monitoring were conducted. The work was focused on the control of melt temperature during casting and the observation of cooling of the metal inside the flask. The simulation was calibrated and was used to simulate the casting process in order to understand the role of parameters on casting quality. Selected casting trials with a variation of process parameters were carried out employing two different alloys and several crucibles and investment materials. This publication is the first part of two papers on platinum investment casting. The second paper deals with the optimization of alloy composition using thermodynamic simulations [5]. The results described in these two papers were presented in an oral presentation at the 28th Santa Fe Symposium ® on Jewelry Manufacturing Technology in 2014 [6].

EXPERIMENTAL

Computational fluid dynamics (CFD) can be applied to simulate the complex flow of the melt during casting and the temperature distribution as a function of time. The methods used in this work are described in [7, 8]. The simulation was conducted in two steps. Form filling during casting was simulated with the software FLOW3D (Version 10.0.3, Flow Science, USA). The temperature distribution after completion of form filling was transferred to the software POLIGON (Version 12.1, Poligonsoft, Russia) in order to simulate the solidification shrinkage and investment heating. Based on the form filling simulation the optimum placement of the parts, the inclination of the sprues and the main sprue diameter were determined. Five different tree designs proposed in [9] were tested for their suitability. One optimized tree was selected for tilt and centrifugal casting experiments, respectively (Figure 1).

In order to set up the simulation of the casting process, materials data for metals and investment materials are required. Relevant material data are the melting range, the fraction of solid, the thermal conductivity, the density and the heat capacity of the alloys as function of temperature. Experimental information of such data is very limited for platinum alloys [10, 11, 12]. Therefore some data have been determined by thermodynamic simulations and in experimental setups at fem [5, 6]. Material data for investment materials

are even more difficult to determine. The thermal expansion, the melting range and the gas permeability have been determined for three selected investment materials [6].
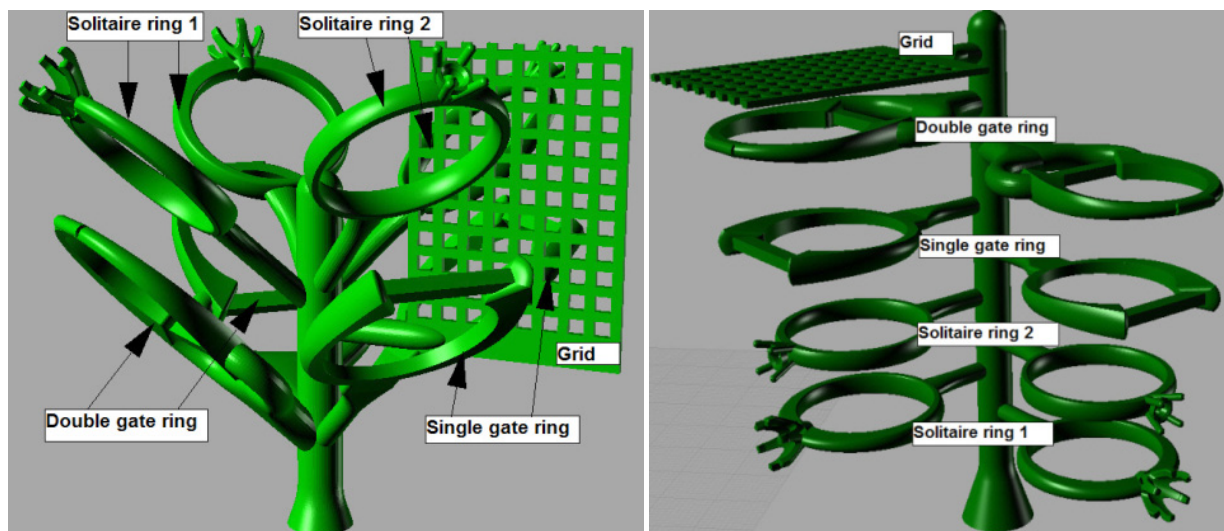

Figure 1: Casting tree setup for tilt casting (left) and centrifugal casting (right). The length of the main sprue was ca. 60 mm.

In order to calibrate the simulation, cooling curves of the metal inside the flask were measured using Type B (Pt30%Rh-Pt6%Rh) thermocouples. Figure 2a shows the thermocouples mounted on the wax tree. The thermocouples were inserted in ceramic tubes and connected to a data logger inside the casting machine (Figure 2b). Again, the setup was adapted to the possibilities given by the specific casting machine. The exact position of the thermocouples was controlled by x-ray imaging of flasks after casting in order to determine the distance of the thermocouples from the tree. This distance was taken into account when calculated and measured cooling curves were compared (Figure 2c). The experimental cooling curves allowed the calibration of the casting simulation for the different type of machines. More details on the experimental setup are given in [13].
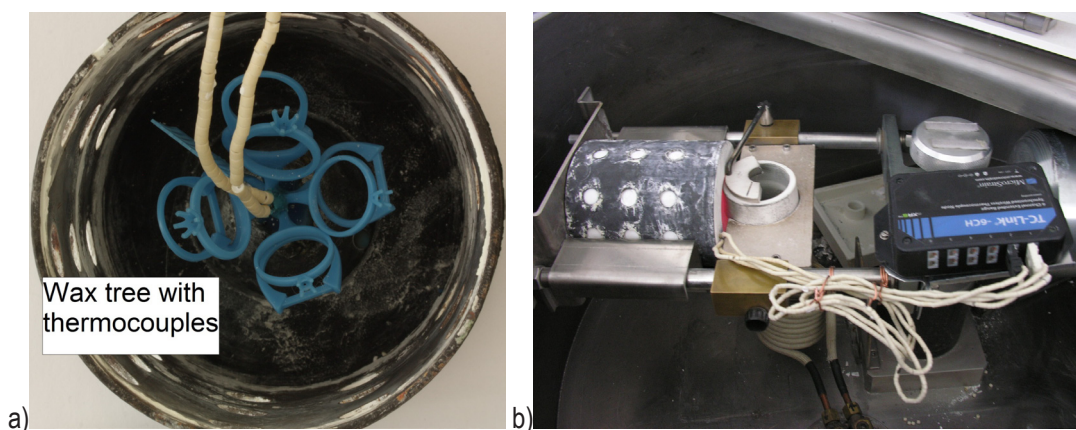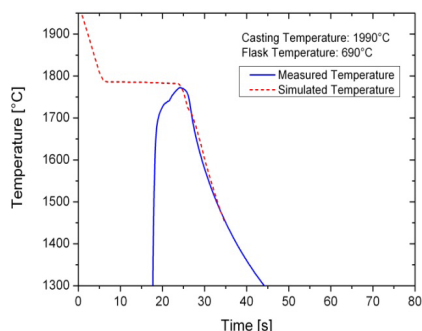
Figure 2: Temperature measurements with thermo couples on the cast tree (a). Flask with the thermo couples and data logger in the centrifugal casting machine (b). Comparison of measured and simulated temperature-time profile (c).

A number of casting experiments was carried out in order to evaluate the influence of the casting parameters on the cast parts. The selection of casting tree design for tilt and centrifugal was made based on the form filling simulation of different tree design variations. Figure 1 shows the two selected tree designs for tilt and for centrifugal casting. Each tree contains 8 typical jewellery pieces and one grid for the determination of form filling. Two pieces of four different rings were used on the tree, in particular "Solitaire Ring 1" for a larger stone, "Solitaire Ring 2" for a smaller stone, a "single gate" ring with one sprue at the ring shank and a "double gate" ring with two sprues at the heavy sections. The two selected trees had a casting weight of ca. 100 g and 125 g for tilt and centrifugal casting, respectively. The casting trials were carried out in purified synthetic air. The tilt casting machine model was a VTC100VTi (Indutherm, Germany). The centrifugal casting machine models were a TCE10 (TopCast, Italy) and a Platincast600 (Linn, Germany). The metal temperature during heating and melting was monitored using a pyrometer and a thermal imaging camera. The experimental setup was slightly different during centrifugal and tilt casting depending on the setup of the machines. The detailed experimental setup is described in [13]. The temperature/time profile in selected areas was recorded for every single casting trial. The "casting temperature" was the temperature of the melt at the moment when it was leaving the crucible. The casting temperatures were 1850°C – 2040°C depending on alloy and casting machine. The flask temperature was varied between 550°C and 950°C. The alloys used were Pt-5Co and Pt-5Ru. In the second part of the work the modified alloy compositions were tested [5]. Eight different investment materials were tested and finally three investment materials were chosen for the casting trials. Pro-HT (Goldstarpowders, UK), referred to as "EBM7" is a one-part jewellery investment material for platinum alloys. R&R Platinum (Dentsply, USA), referred to as "EBM8" is a two-part jewellery investment material for platinum alloys. Sherafina 2000 (Shera Werkstofftechnologie, Germany), referred to as "EBM5" is a dental investment.

After casting the tree was water-blasted. In order to evaluate the surface quality its surface was inspected by optical microscopy and scanning electron microscopy (SEM). The form filling was assessed by the form filling of a grid. In order to identify shrinkage porosity and other casting defects X-ray computer tomography and metallographic investigation were carried out.

CASTING RESULTS

FORM FILLING

The form filling ability was assessed by determining assessing the percentage of fully intact grid parts (Figure 1) and strongly depends on the alloy composition. In previous studies with a "diabolo-type" tree, Pt-5Co showed superior form filling in comparison to Pt-5Ru [1]. This was confirmed for the more conventional trees employed during the present study (Table I). Centrifugal casting of Pt-5Co allowed complete form filling at melt and flask temperatures exceeding 1850°C and 550°C, respectively.

The casting temperature of Pt-5Ru was required to be approx. 100 K higher. Tilt casting generally required higher casting temperatures for complete form filling compared to centrifugal casting. In case of Pt-5Co, it was sufficient to increase the casting temperature by approx. 100 K. For Pt-5Ru not only the casting temperature but also the flask temperature had to be increased by 100 K, respectively. The reasons for the poorer form filling ability of Pt-5Ru are explained in detail in [5].


Table I: Grid filling as function of casting and flask temperature for centrifugal and tilt casting of Pt-5Ru and Pt-5Co.

Centrifugal casting		
Casting temperature °C/ Flask temperature °C	Alloy	Grid filling in %
1850/850	Pt-5Ru	28
1950/850	Pt-5Ru	100
1850/850	Pt-5Co	100
1850/550	Pt-5Co	100
Tilt casting		

1950/850	Pt-5Ru	6
1950/950	Pt-5Ru	62
2040/950	Pt-5Ru	100
1825/850	Pt-5Co	20
1950/850	Pt-5Co	100

SURFACE QUALITY

The surface quality depends primarily on the alloy and on the investment material. For Pt-5Ru significant differences were also observed between thin and thick section of the same cast part (Figure 3). With the investments EBM5 and EBM7, the surface quality of Pt-5Ru is very poor in the thick sections. It deteriorates with increasing casting and flask temperature for every investment material; however the extent to which this effect occurs is different for each investment material. EBM8 shows only a slight deterioration of the surface even at high casting and flask temperature (Figure 3e). In comparison, the surface quality of EBM7 was very poor in the thick sections and showed a glassy layer at higher magnification, while it had acceptable surface quality in the thin sections (Figure 3a). The SEM image (Figure 3b) reveals dimples on the surface, which are formed by gas bubbles at the interface of metal and investment (Figure 3c). The investment material was molten in contact with the metal during casting and the gas bubbles are presumed to form due to the coagulation of the investment porosity. The molten area appears to be covered by a transparent and shiny surface layer similar to the appearance of silica glass. The glassy layer is about 500 µm thick. An XRD measurement of molten investment at the interface (Figure 3d) indicates its amorphous structure, which can be seen as an evidence for its glassy nature. Prior to the casting experiment, the investment material consisted of hexagonal α–quartz, tetragonal β-cristobalite, orthogonal Tridymite and $Mg_3(PO_4)_2$. Apparently, the melting point of EBM7 is too low to withstand the high temperatures of the Pt-5Ru melt. EBM8 on the other hand shows a smooth interface of the complete double gate ring (Figure 3e, f). No gas pores are observed at the interface and the investment was not molten during casting (Figure 3g). A molten surface layer was not observed, only a ca. 30 µm sintered layer. Therefore, the investment still shows its crystalline structure of pure hexagonal α–quartz (Figure 3h).
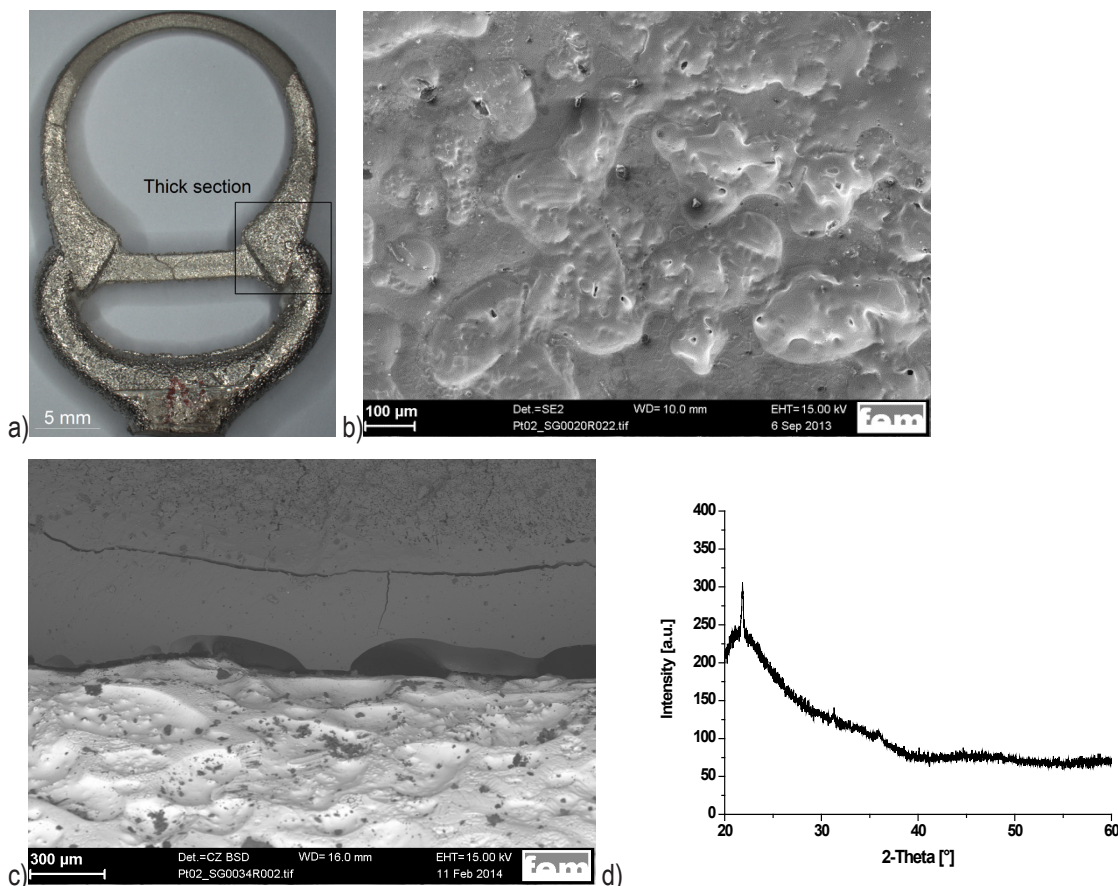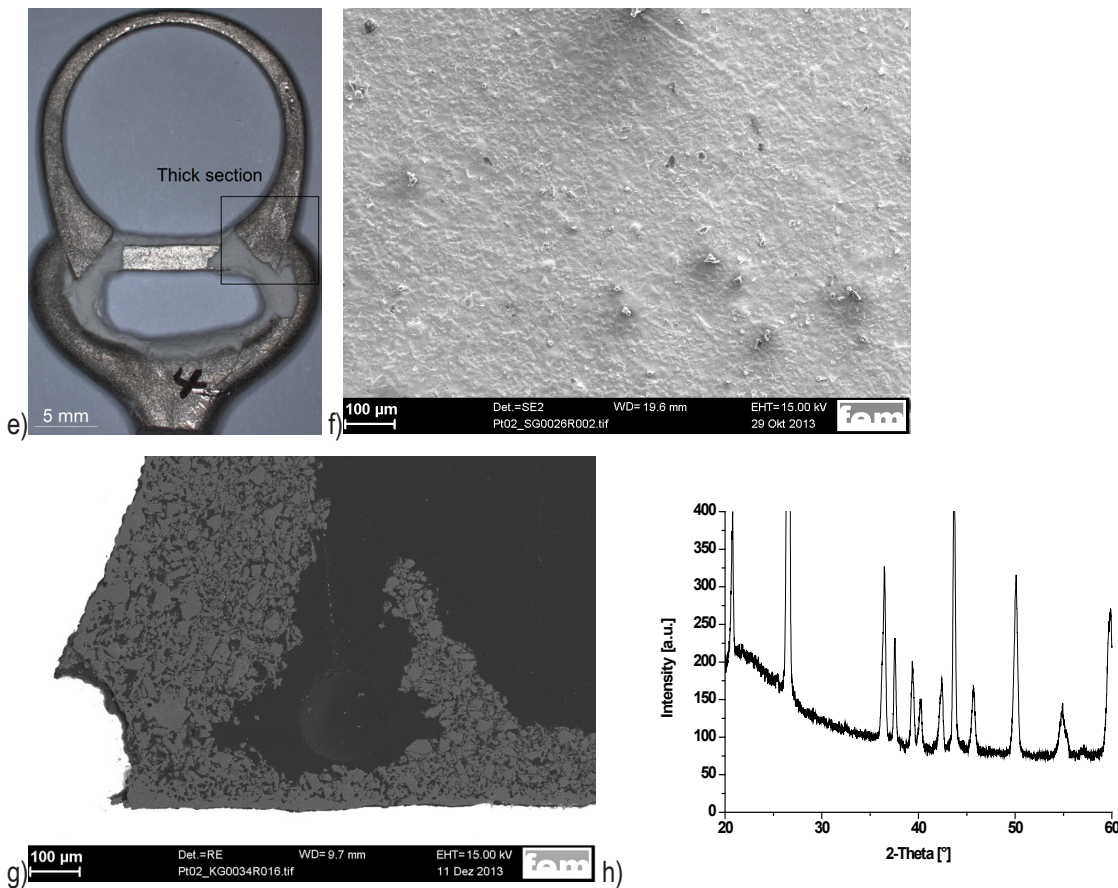
Figure 3: Influence of investment material on surface quality of Pt-5Ru. T_{melt} = 1850°C / T_{flask} = 850°C. EBM7 (a-d) and EBM 8 (e-h). Optical microscope image (a, e). SEM surface image of thick section (b, f). SEM image of the metal-investment interface in the thick section (c, g). XRD measurement of the investment at the interface (d, h).

The dental investment EBM5 showed similar characteristics as EBM7, but had slightly better surface quality. In an isothermal melting test (10 min) none of the investments did melt at 1675°C (solidification temperature of Pt-5Co). At 1725°C EBM5 and 7 partially melted, while EBM8 did not melt. At 1775°C (solidification temperature of Pt-5Ru) all investments were melting. It appears that EBM8 has the highest temperature stability and withstands melting at the short term heating during casting. In case of Pt-5Co, the casting and flask temperatures have very little influence on the surface quality. With all three investment materials a blue layer of cobalt silicate forms, but the surface is generally smooth.

POROSITY

The casting simulation predicts decreasing shrinkage porosity with increasing casting and flask temperature. Metallographic investigations confirm this prediction for both casting machines in agreement with further studies [1]. In general, large shrinkage porosity has to be avoided by proper sprues like it is the case for the double-gate ring. In that case defect-free castings can be achieved with Pt-5Co. Only some very small (<1 µm) and scattered gas pores and oxide inclusions were found, which do not deteriorate casting quality. Pt-5Ru on the other hand, shows a pronounced tendency to form micro shrinkage porosity, even for proper sprues. An explanation of this effect is given in [5]. A remedy to close the micro shrinkage pores is hot isostatic pressing as described in [14]. In tilt casting the tendency for micro shrinkage porosity is less pronounced in comparison to centrifugal casting (Figure 4a and b). However, very high casting (2040°C) and flask temperatures (950°C) are required for complete form filling of filigree items. As described above, very temperature resistant investment materials are required for good surface quality. Generally, the investment material has a rather small influence on the porosity formation, although EBM8 shows slightly better results compared to EBM5 and EBM7.
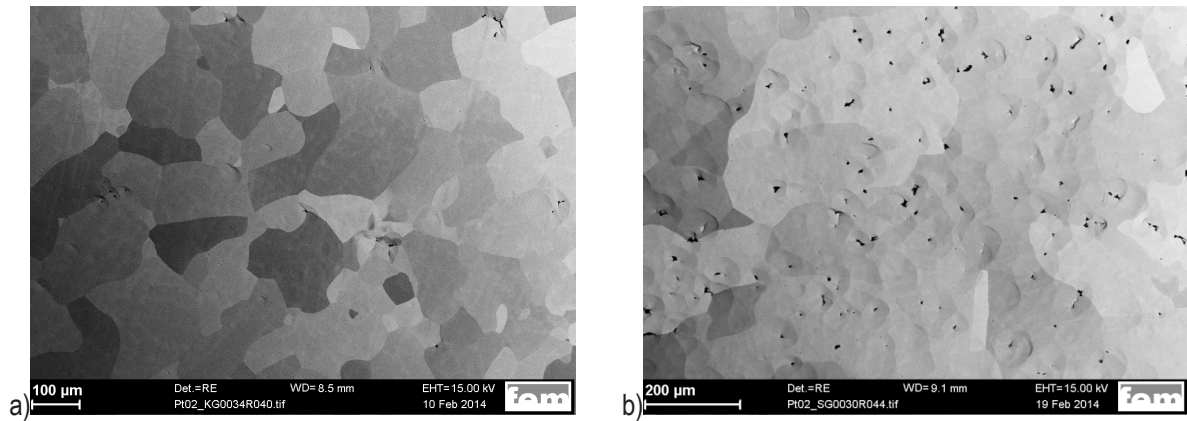
Figure 4: Microstructure and porosity of Pt-5Ru in tilt casting (a) and centrifugal casting (b).

DISCUSSION

Casting simulation is a powerful tool for the prediction and understanding of the defects in casting. Casting can be understood as a two-step process. The first step in casting is the form filling process. During this step the forces acting on the melt are controlling its flow into the flask. In tilt casting gravity force is the major acting force. Therefore the flow speed of the melt is relatively low resulting in a comparatively long form filling time promoting significant cooling during form filling. For the chosen tree the temperature of the melt significantly deviated along the tree axis resulting in a temperature gradient from the main sprue to the tree tip. The temperature difference of the melt entering the sprue in the first layer of parts and in the second layer was about 40 K (Figure 5a). The complete filling of the tree takes about 0,6 s. The filling time is approx. 50% shorter in centrifugal casting compared to tilt casting although the tree is ca. 25% heavier. The shorter filling time results in a higher residual melt temperature, which explains the better filling ability of centrifugal casting at low casting and flask temperature. Based on the filling simulation the tree geometry was optimized. The main sprue diameter in tilt casting could be reduced from 10 mm to 5 mm, because the filling speed increases for the smaller main sprue. In addition, this saves about 50% of the tree weight – an important amount of otherwise ineffectively used precious metal.

In centrifugal casting, the main force acting is the centrifugal force. The way how the tree is filled depends strongly on the mounting of the pieces on the main sprue. If the parts are mounted on the leading side of rotation, the inertia causes an acceleration of the melt to the trailing side of the main sprue. Parts that are mounted under an angle of 90° to the main sprue are then successively filled from the tip of the main sprue towards the ingate. This situation is illustrated in Figure 5b for the upper row of parts. If the parts are mounted on the trailing side of the tree, the momentum of inertia of the melt causes all parts to be filled simultaneously. The simulation indicates that such simultaneous filling might cause cold shuts. This effect, however, is substantially smaller because the acting forces are small.
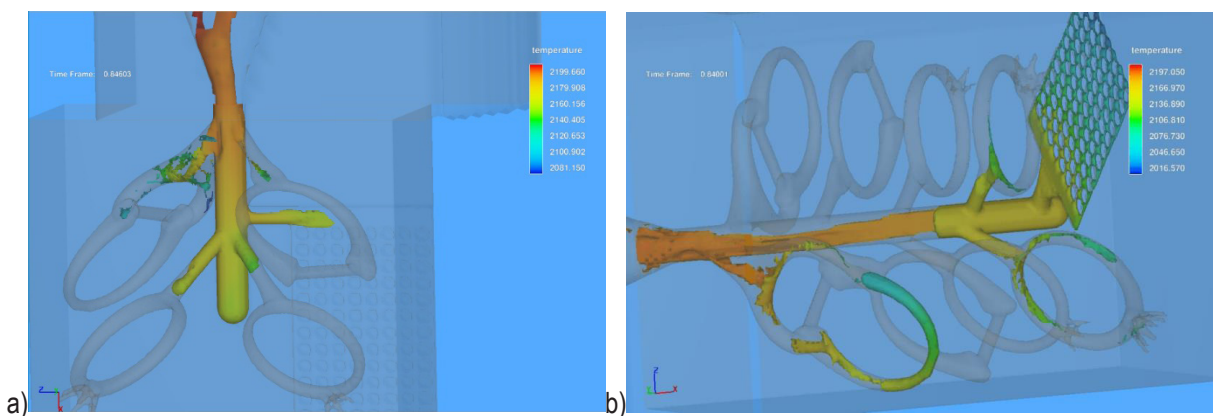

Figure 5: Form filling simulation for tilt casting (a) and centrifugal casting (b).

The second casting step, the solidification, was simulated using the software Poligon that allows simulating the solidification shrinkage and therefore local shrinkage porosity in the parts (Figure 6). The red areas indicate high shrinkage porosity. At the tip of the tree nearly no porosity should be observed in the cast parts according to the simulation. The single gate appears being most prone to shrinkage porosity, especially at its thick section that is probably isolated from the melt in the main sprue during solidification due to a premature solidification in the tighter parts of the sprue. In this case, feeding with fresh melt is interrupted. The pressure gradient inside the part forces the remaining melt towards the tip of the tree.

The heating of the investment was also simulated using Poligon. Figure 7 shows the temperature distribution in the investment for the double-gate. Along the thin ring shank the temperatures are relatively low. Critical positions are the thick sections of ring. A hot spot is found where the heavy sprues are connected to the ring. With increasing casting and flask temperature the local overheating increases significantly. Figure 3c and g show the investment reactions at the thick sections. The locally very high temperature causes the breakdown of some less stable investment materials, which results in poor surface quality. The breakdown of the investment materials is promoted by high amounts of magnesium phosphate that is used as a sintering additive, which reduces the melting range of the investment (15). Such additives can be a significant restriction for the investment material. In order to avoid investment breakdown more stable investment materials have to be used or the alloy might be modified in the solidification temperature as described in [5].
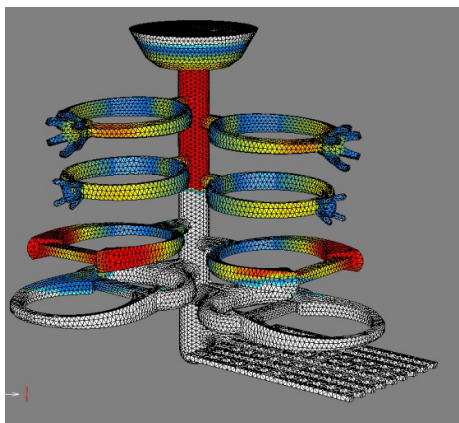

Figure 6: Shrinkage porosity in centrifugal casting obtained by Poligon. Red areas indicate a high probability for porosity, while grey areas should be free of pores.
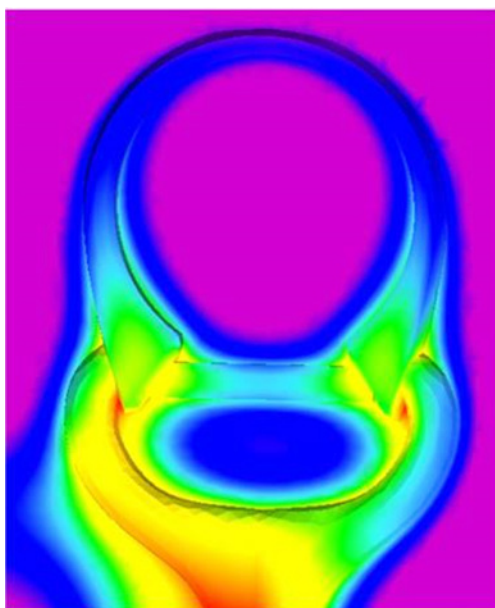

Figure 7: Temperature distribution in the investment after centrifugal casting of PtCo5 obtained by Poligon (T_{flask} = 850°C).

SUMMARY

Pt-5Co has a good form filling compared to Pt-5Ru independently of flask and casting temperature. For the machines used for the duration of this study, form filling was better in centrifugal casting compared to tilt casting, because the acting forces are 4 times higher. Hence, in centrifugal casting the filling was faster and the melt temperature remained higher during filling. As a consequence, higher overheating was needed in tilt casting to obtain the same filling.

An improvement of the mold filling of Pt-5Ru can be achieved with increased casting and flask temperature. However, very stable investment materials have to be used in order to avoid investment breakdown. Locally, very high investment temperatures can occur as demonstrated by the simulation. Less stable investment materials might locally melt or sinter, which results in poor surface quality. The most stable investment material in this study contained high amounts of hexagonal α-Quartz and low amounts of sintering additives. As predicted by casting simulation, the shrinkage porosity decreases with increasing casting and flask temperatures. This was particularly true for Pt-5Co, which only showed very small gas pores and oxide inclusions und optimum conditions. Pt-5Ru showed a pronounced tendency to form micro shrinkage that can be hardly avoided in centrifugal casting. In tilt casting, the micro shrinkage was less pronounced due the slower cooling in the specific machine employed in this work. The project showed the limits of conventional jewellery alloys (Pt-5Ru and Pt-5Co). A further alloy optimization appears necessary [5].

Important and helpful indications about the tree design, positioning or inclination of the parts and of the tree and the temperature distribution of the investment could be concluded from the casting simulation. Particularly in the case of centrifugal casting (where the acting forces are more complex), the casting simulation helps understanding casting defects and finding suitable solutions.

REFERENCES

[1]   U.E. Klotz and T. Drago, 'The role of process parameters in platinum casting', Platinum Metals Rev. 2011, 55, (1), 20-27.

[2]   D. Miller, T. Keraan, P. Park-Ross, V. Husemeyer and C. Lang, "Casting platinum jewellery alloys, The effects of composition on microstructure", Platinum Metals Rev., 2005, 49, (3), 110–117

[3]   D. Miller, T. Keraan, P. Park-Ross, V. Husemeyer, A. Brey, I. Khan and C. Lang, "Casting platinum jewellery alloys, Part II: The effects of casting variables on fill and porosity", Platinum Metals Rev., 2005, 49, (4), 174–182

[4]   T. Fryé and J. Fischer-Buehner, ,Platinum alloys in the 21st Century: a comparative study', in "The Santa Fe Symposium on Jewelry Manufacturing Technology 2011", ed. E. Bell, Proceedings of the 25th Symposium in Albuquerque, New Mexico, USA, 15th-18th May 2011, Met-Chem Research Inc, Albuquerque, New Mexico, USA, 2011, pp. 201-230.

[5]   U.E. Klotz, T. Heiss and D. Tiberto, "Platinum investment casting, Part II: Alloy optimization by thermodynamic simulations and experimental verification", Johnson Matthey Technology Rev. submitted 2014

[6]   U.E. Klotz, T. Heiss, D. Tiberto, F. Held, "Platinum investment casting - Material properties, casting simulation and optimum process parameters", in "The Santa Fe Symposium on Jewelry Manufacturing Technology 2015", ed. E. Bell and J. Haldeman, Proceedings of the 29th Symposium in Albuquerque, New Mexico, USA, 17th-20th May 2015, Met-Chem Research Inc, Albuquerque, New Mexico, USA, 2015, in press.

[7]   D. Tiberto and U. E. Klotz: 'Computer simulation applied to jewellery casting: challenges, results and future possibilities', MCWASP XIII: International conference on modelling of casting and advanced solidification processes, Schladming, Austria, 17-22 June 2012, 2012, IOP Publishing.

[8]   U. E. Klotz and D. Tiberto: 'Computer simulation in jewelry technology - meaningful use and limitations', The 26th Santa Fe Symposium of Jewelry technology, Albuquerque, NM, USA, 20-23 May 2012, 2012, Met-Chem Research, Albuquerque, NM, USA, 297-320.

[9]   J. Maerz, 'Platinum casting tree design', in "The Santa Fe Symposium on Jewelry Manufacturing Technology 2007", ed. E. Bell, Proceedings of the 21st Symposium in Albuquerque, New Mexico,, USA, 22nd-25th May, 2007, Met-Chem Research Inc, Albuquerque, New Mexico, USA, 2007, pp. 305-322.

[10]   G. Beck, 'Edelmetall-Taschenbuch'; 1995, Degussa AG, Frankfurt und Hüthig GmbH, Heidelberg.

[11]   S. Mehmood, U. E. Klotz, and G. Pottlacher, Met. Mat. Trans A, 2012, 43(13), 5029-5037.

[12]   S. Mehmood, U.E. Klotz, G. Pottlacher, "Thermophysical properties of the platinum-copper system", EPD Congress 2011 TMS Annual Meeting, San Diego, USA 27th Feb – 3rd Mar 2011, Volume 1, 2011, 167-173

[13]   U.E. Klotz, T. Heiss and D. Tiberto, 'Entwicklung der Gießsimulation und experimentelle Untersuchungen von Platin legierungen zur Optimierung des Feingussprozesses für Uhren- und Schmuckanwendungen' (in German), Final report of the research project AiF-IGF 16413N, 2014, available on request at www.fem-online.de

[14]   T. Fryé, J.T. Strauss, J. Fischer-Buehner, U.E. Klotz, ,The effects of hot isostatic pressing of platinum alloy cast ings on mechanical properties and microstructures', in "The Santa Fe Symposium on Jewelry Manufacturing Technology 2014", ed. E. Bell and J. Haldeman, Proceedings of the 28th Symposium in Albuquerque, New Mexico, USA, 18th-21st May 2014, Met-Chem Research Inc, Albuquerque, New Mexico, USA, 2014, pp. 189-210.

[15]   H. Gamsjäger, 'Encyclopedia of Earth Science' Springer 1998, pp. 582-587