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Teresa Frye is the owner and President of TechForm Advanced Casting Technology. A leading expert on jewelry casting and a prolific researcher, she has published technical papers and articles worldwide, and speaks frequently at industry conferences in the United States and Europe. TechForm provides platinum castings to a broad customer base in the United States, including many of the top jewelry brands. Her publications have appeared in Platinum Metals Review, The Johnson Matthey Technology Review, The Santa Fe Symposium on Jewelry Manufacturing Technology, The Jewelry Technology Forum, the MJSA Journal, and JCK Magazine, among others

In earlier research a better understanding of the solidification characteristics for a number of platinum-based alloys was established. The findings demonstrated a strong tendency toward the formation of shrinkage and gas porosity upon solidification, and hot isostatic pressing (HIP), a high-pressure thermal treatment developed as a densification process, was proven to be an effective method to minimize and/or eliminate this porosity.
While the previous results made it clear that porosity had been significantly reduced following the HIP process, the authors had not yet explored the full range of HIP's effects in terms of post-processed microstructure and mechanical properties. The goal of this new phase of research is to further our understanding by characterizing the post-HIP effects on platinum based castings with respect to grain size and shape, chemical distribution, and mechanical strength.

The Effects of Hot Isostatic Pressing of Platinum Alloy Castings on Mechanical Properties and Microstructures

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PREVIOUS RESEARCH

In the earlier work by Fryé and Fischer-Bühner , results on solidification behaviors of several platinum-based alloys were reported. The complete study explored variations in solidification patterns that occur with different alloys, sprue systems, casting atmospheres, and investments.

To briefly recap this work, Figures 1a & 1b show longitudinal cross-sections of 95Pt5Ru at 50X magnification, revealing the interior as-cast quality. Both sections are characterized by significant amounts of shrinkage and gas porosity, demonstrating the challenge in achieving fully dense castings in this alloy. Even with the use of optimized feed sprues (Figure 1b) porosity levels remained high with significant room for improvement. In contrast, Figure 1c shows a cross-section of the same sample type that has been hot isostatically pressed. This casting became fully dense following HIP, demonstrating the efficacy of this process in closing up sub-surface porosity.

It is important to note that alloy type was proven to affect levels of porosity quite significantly. Porosity formation upon solidification was unique for all alloys tested, with 95Pt5Co behaving most favorably and 95Pt5Ru the least. Regardless of alloy or casting conditions, all of the samples evaluated resulted in as-cast product that exhibited sub-surface shrinkage and gas porosity that would likely require some degree of rework to yield an optimal product.
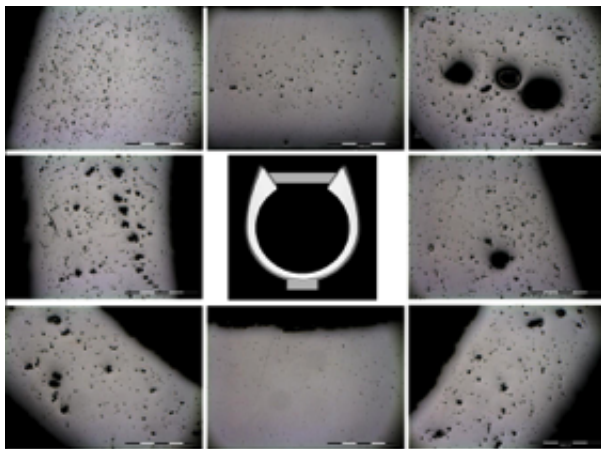

Figure 1a - 95Pt5Ru
Single bottom sprue
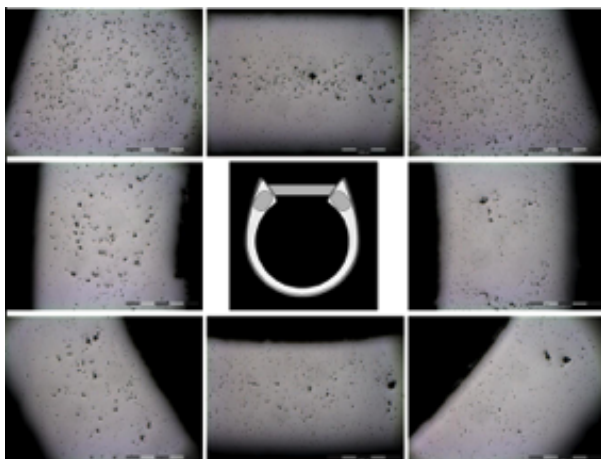Shrinkage/gas porosity prevalent due to inadequate molten feed

Figure 1b - 95Pt5Ru
Double top sprue
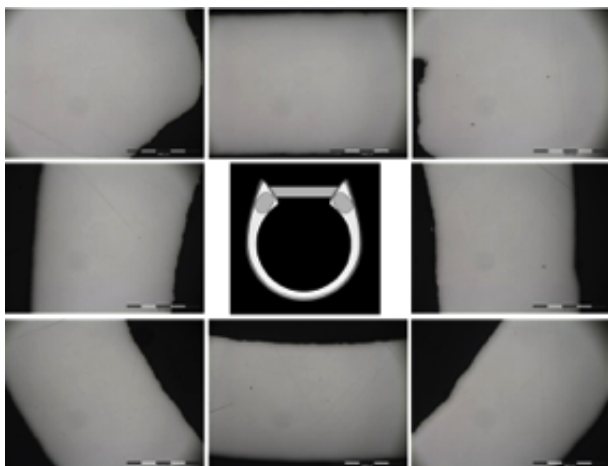Small, scattered microshrinkage and gas porosity remain despite use of heavier spruing

Figure 1c - 95Pt5Ru
Double top sprue with HIP
Microshrinkage has been eliminated through diffusion bonding resulting from the HIP process

Key conclusions from the 2011 study can be summarized as follows:
1) A single sprue approach as shown in Figure 1a was not recommended for the geometry tested because the multiple sprue approach alone demonstrated a significant reduction in porosity for all alloys tested.
2) Even with use of an optimized and directionally solidified sprue system, none of the alloys were capable of achieving fully dense castings.
3) HIP was proven as an effective means to eliminate or nearly eliminate the presence of porosity in all platinum alloys tested.

OVERVIEW OF HOT ISOSTATIC PRESSING

Companies that build HIP equipment or perform the HIP process describe an isostatic press as something that forms and densifies powdered and cast materials using liquid or gas under extremely high pressure. Unlike mechanical force which compresses a workpiece from one or two sides, the isostatic pressure is applied uniformly on all sides of an object, eliminating internal porosity without changing net shape. Typical product improvements cited by the HIP industry are the elimination of internal voids, improvements in product consistency, and improvements in the soundness and mechanical properties of materials. The fundamental material change underlying these improvements is the attainment of a higher density material in comparison with its pre-HIP condition. Indeed, HIP is well-known for its ability to densify and improve properties for a variety of materials including metal castings, composites, polymers and parts made from ceramic or metal powders.

The isostatic nature of pressure in the HIP process as described above is key to maintaining the dimensional integrity of a casting during HIP; the pressure being equal on all sides lends to a uniform compression of the material with product dimensions typically remaining intact. Although, as seen in Figure 2, local deformations in the form of "dimpling" can occur when the sizes of internal pores are extremely large and diffusion bonding collapses exterior surfaces inward. This difficult to feed thick-to-thin geometry for the channel band represents an extreme example of subsurface shrinkage porosity and is a powerful representation of the pore collapsing that occurs with HIP. However, one can certainly argue the point that internal porosity is eliminated without changing net shape based on these images. Aside from these obvious deformations, detailed dimensional studies are required to determine more clearly what the level of impact is to casting dimensions in platinum-based alloys.
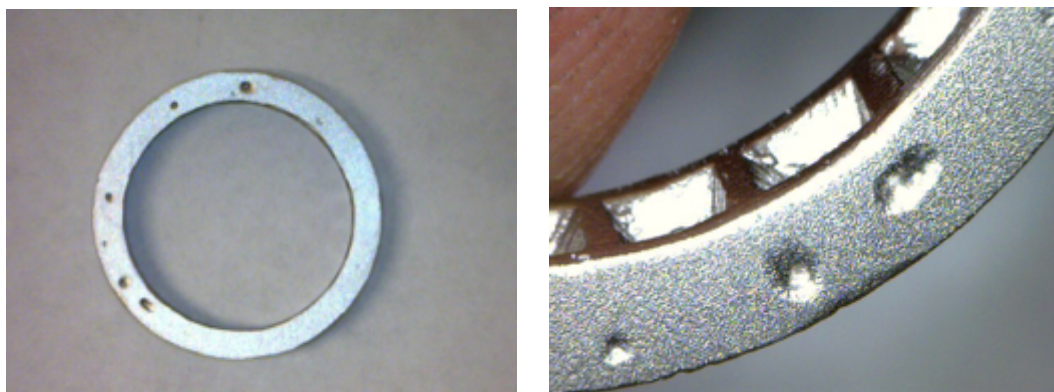

Figure 2   HIP dimples in 95Pt5Ru channel band

To better demonstrate how the HIP process works, Figure 3 shows the schematic for a typical HIP unit. The unit contains a high temperature furnace enclosed in a pressure vessel. Parts are typically placed within the chamber in vertical layers with the use of steel or ceramic shelving to maximize load capacity. During operation the HIP chamber is first placed under vacuum, followed by flooding with an inert gas, usually argon, which is used to apply the isostatic pressure. The temperature and pressure is then ramped up and left to dwell for a specified period of time depending upon the material's properties. Parts become densified when the material's yield strength is surpassed, creating a plastic flow that forces internal voids to collapse under differential pressure. The internal surfaces of the voids diffusion bond together, increasing density and thereby improving the material properties. HIP unit sizes span from small laboratory size up to large-scale industrial. The unit shown in Figure 4 is an example of a smaller scale unit.
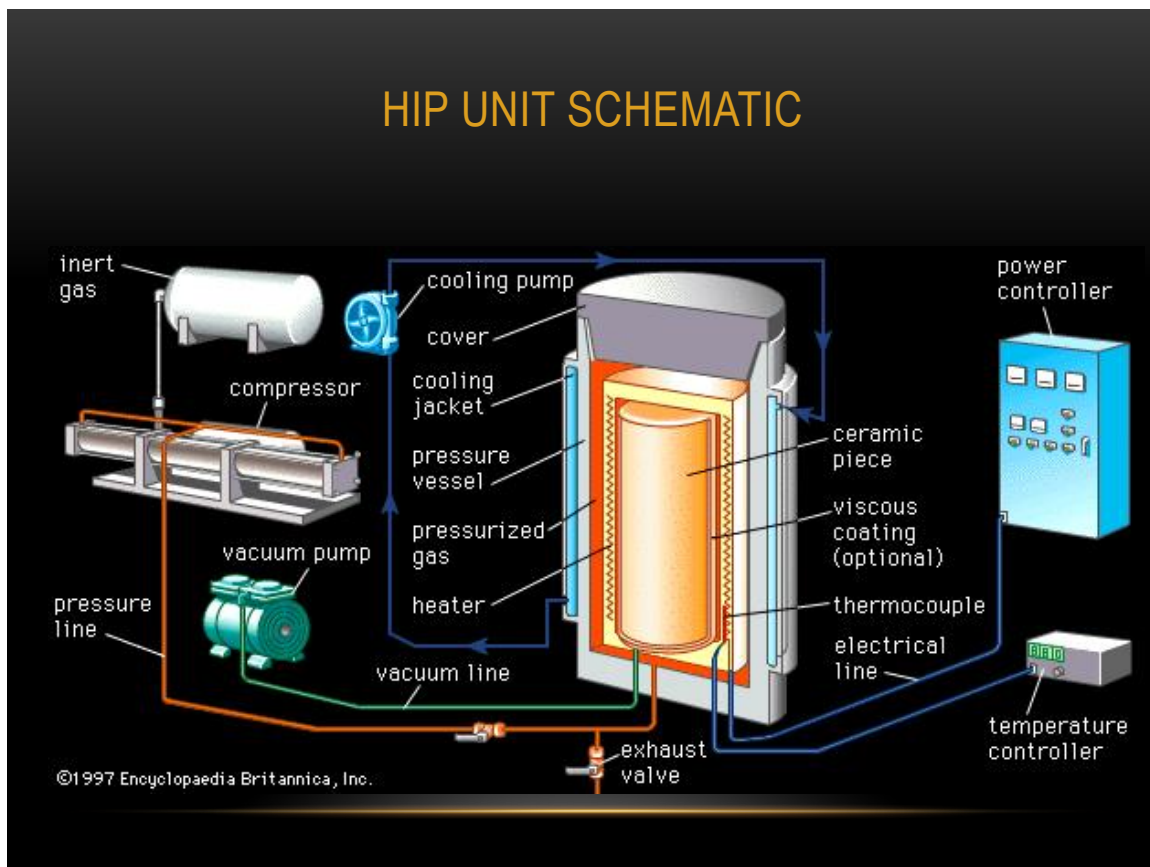

Figure 3   Schematic of typical HIP unit

Figure 4 HIP unit, courtesy Avure Technologies

Not all metals will HIP effectively and the extent to which an alloy will respond to HIP is a function of its creep resistance. Creep is a solid material's tendency to move slowly and deform permanently under stress. In metals, creep increases with temperature and starts at approximately 40 to 50% of an alloy's melt temperature.

The rate of creep is a function of temperature, the material's properties, and the amount of pressure that is applied. In order to achieve optimum material properties, the parameters used in a HIP cycle must be precisely dialed in according to the needs of the alloy. Typical parameters including both metals and ceramics will generally fall into the ranges shown in Table 1:

Table 1 –HIP Parameter Ranges, courtesy Avure Technologies

Parameter	Typical Lower Limit	Typical Upper Limit
Temperature	500°C	1400°C
Pressure	7,000 PSI	45,000 PSI
Dwell Time	2 hours	4 hours
Cooling Rate	1°C per minute	100°C per minute

PHASE II RESEARCH: COMPARATIVE STUDY OF THE EFFECTS OF HIP ON MICROSTRUCTURE AND MECHANICAL PROPERTIES

The goal of this phase of research was to characterize post-HIP effects on castings with respect to grain size and shape, chemical distribution, and mechanical strength. The following sections report our methods, results and conclusions.

Test Geometry

A tapered test specimen was chosen to assess microscopic porosity levels, density, and hardness before and after HIP. As shown in Figure 5, the test specimen was designed to promote directional solidification with a single heavy sprue attached to the thickest end.
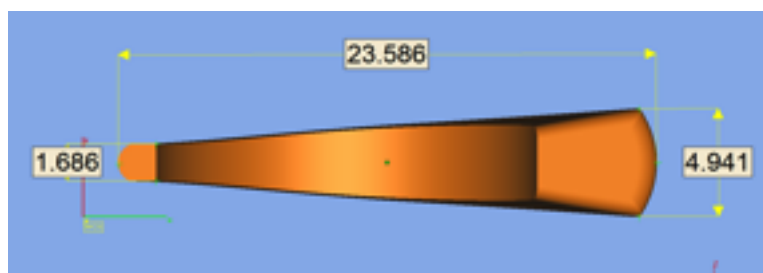

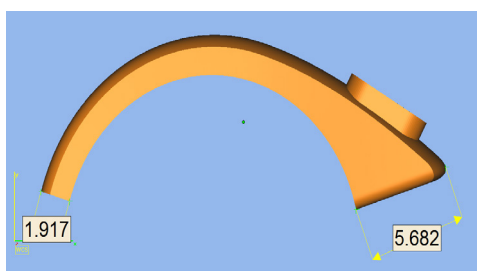Figure 5 Tapered test specimen (units in mm)

Figure 5 Tapered test specimen (units in mm)

The casting parameters and conditions for our trials are shown in Table 2. Standard pour temperatures, flask temperatures and firing curves were used. The flasks were cast using a centrifugal casting machine with induction heating and each tree contained two test geometries for each alloy. One casting was retained for as-cast sampling and the second casting was HIPed according to parameters specific for platinum alloys but proprietary to the HIP vendor.

Table 2 Casting Parameters

Alloy	Pour Temp C	Flask Temp C	Casting Condition
95Pt5Ru	1870	850	1 As-cast; 1 HIPed
90Pt10Ir	1870	850	1 As-cast; 1 HIPed
90Pt10Rh	1960	850	1 As-cast; 1 HIPed
95Pt5Co	1850	850	1 As-cast; 1 HIPed
1) All patterns 3D printed for dimensional precision			
2) All trees were identically assembled with 2 samples per tree			
3) All flasks air cooled identically			
4) All alloys were HIPed in the same load			

RESULTS I: EFFECTS OF HIP ON MICROSTRUCTURE

Given the high levels of porosity seen in 95Pt5Ru and the relatively low levels seen in Pt95Co5, these two alloys were chosen for our report on microstructural changes brought about by the HIP process. Figure 6 demonstrates significant porosity levels in the as-cast state of 95Pt5Ru. The pores in this alloy are interdendritic microshrinkage pores; such pores form during the spontaneous solidification of the alloy that occurs so rapidly that continued feeding is not possible. The HIP process has successfully closed these microshrinkage pores, such that the microstructure is completely dense after HIPing.
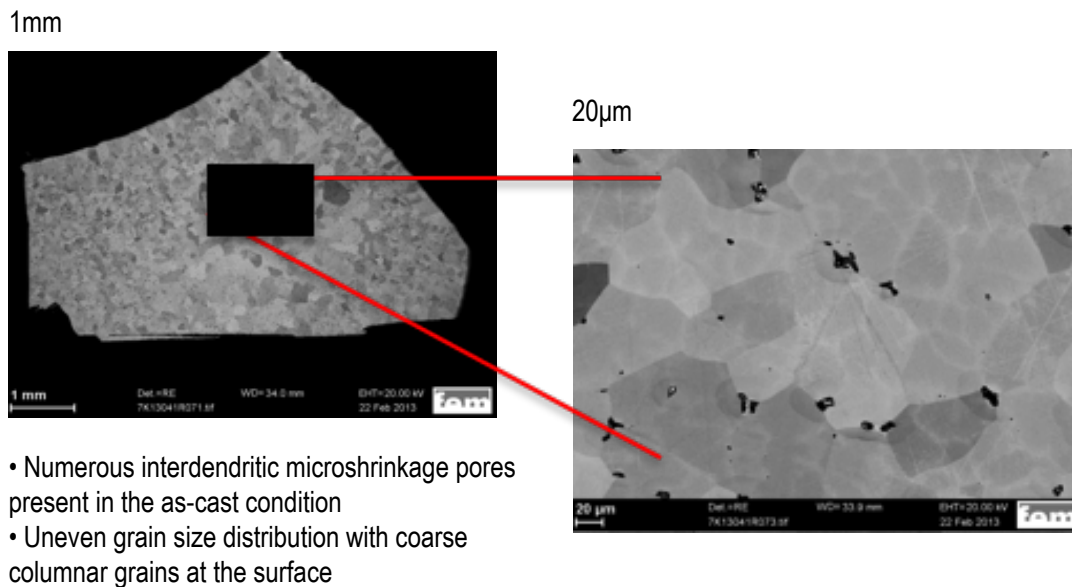
1mm

20µm

• Numerous interdendritic microshrinkage pores present in the as-cast condition
• Uneven grain size distribution with coarse columnar grains at the surface

Figure 6 - 95Pt5Ru As-cast

1mm

20µm

• Dense and pore-free microstructure
• Even grain-size distribution
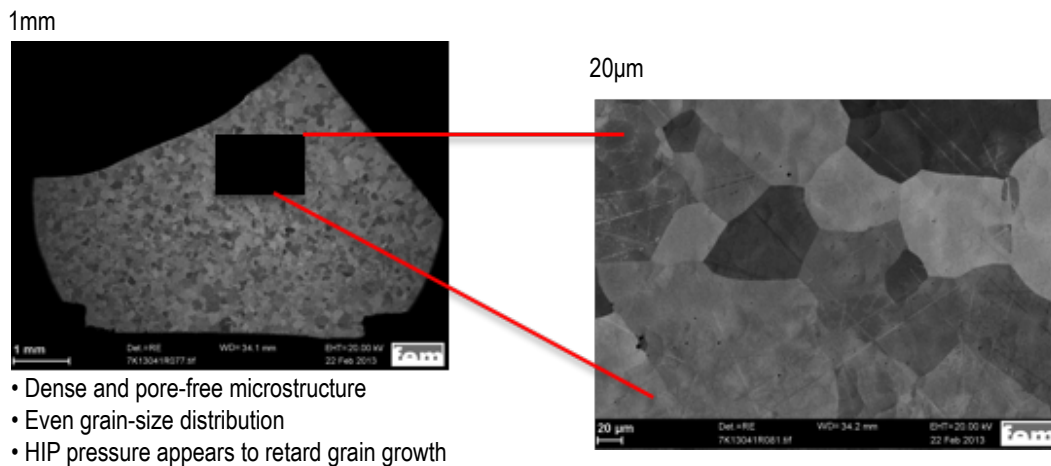• HIP pressure appears to retard grain growth

Figure 7 - 95Pt5Ru HIPed

Another important finding is that grain size is not negatively affected by the HIP process (Figure 7). A simple heat treatment to the same thermal parameters as the HIP processing (without the use of pressure) is neither capable of fully closing the pores from the as-cast condition, nor maintaining grain size (Figure 8). While the amount and the size of pores are clearly reduced, grains are growing substantially during heat treatment. Thus, any beneficial effect of porosity reduction is compromised by grain growth. Based on this result, it would appear that the pressure used in the HIP process has the added benefit of retarding grain growth. Figure 9 demonstrates the comparative grain sizes of 950PtRu in the as-cast, HIPed and heat-treated conditions.

1mm

30μ

• Heat treatment same as HIP thermal curve
• Reduction of microshrinkage porosity through heat only
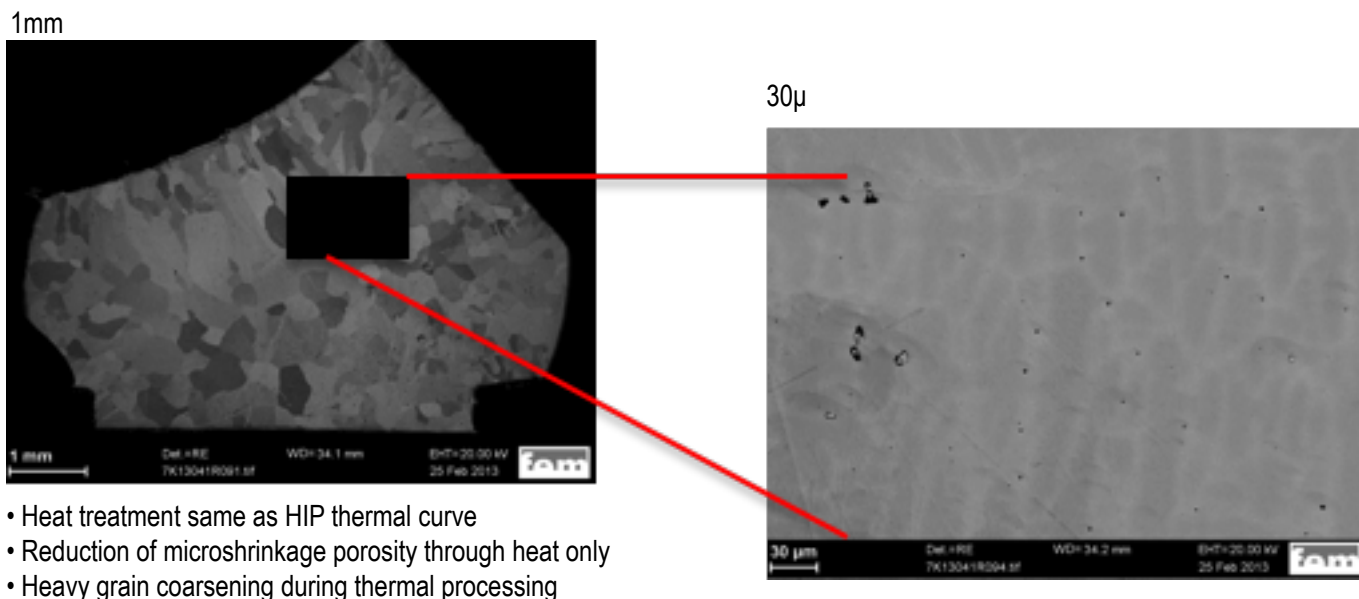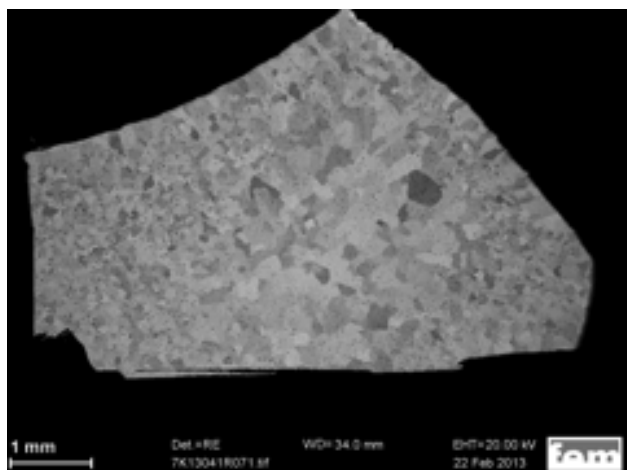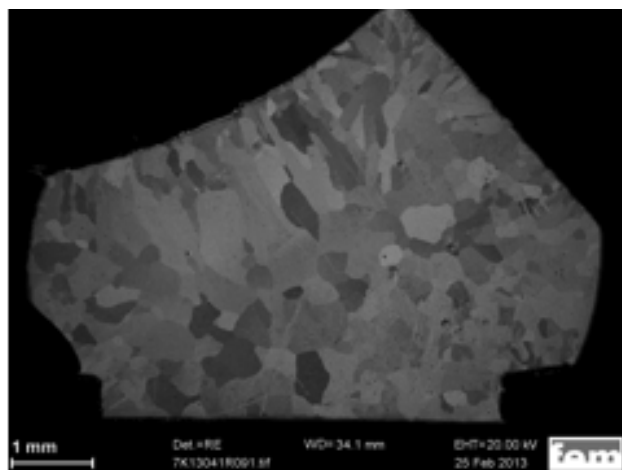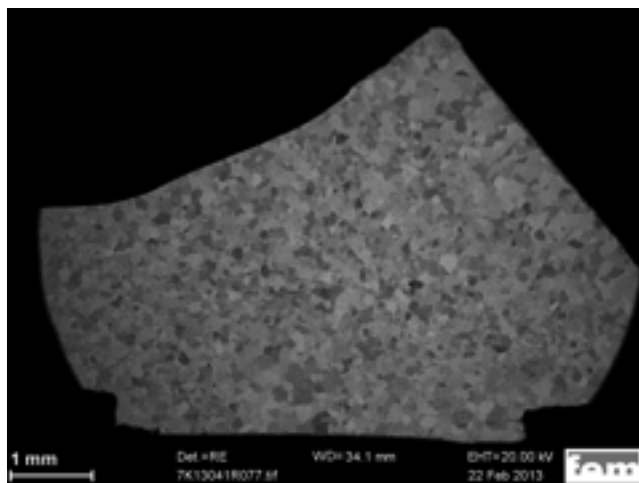• Heavy grain coarsening during thermal processing

Figure 8 - 95Pt5Ru heat treated only

As cast

Heat treated

HIPed

Figure 9 - Comparative microstructures 95Pt5Ru

The casting sample of 95Pt5Co shows no visible macroscopic or microscopic porosity in the as-cast condition (Figure10). However, compared to 95Pt5Ru the grains are extremely large. Their size and shape indicates a relatively slow solidification process where a few grains were nucleated at the surface of the part, which then grew into the center. As a consequence, there was sufficient time for feeding and the microstructure is free of pores. Therefore, during HIPing few changes of the microstructure occurred in the 95Pt5Co (Figure 11). This is not to say that HIP does not provide any benefit to 95Pt5Co castings. The previous research on larger samples demonstrated a tendency of this alloy to form large gas pores and centerline shrinkage porosity that were either eliminated or reduced in size by the HIP process.
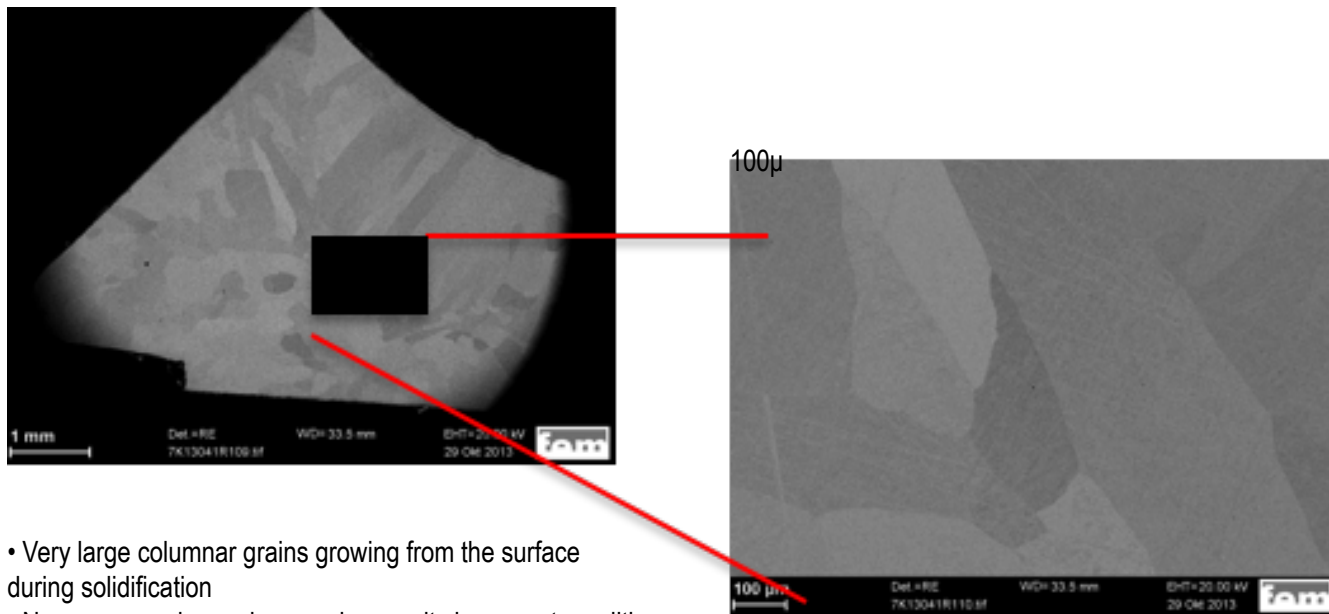

• Very large columnar grains growing from the surface during solidification
• No macroscopic or microscopic porosity in as-cast condition

Figure 10 - 95Pt5Co As-cast

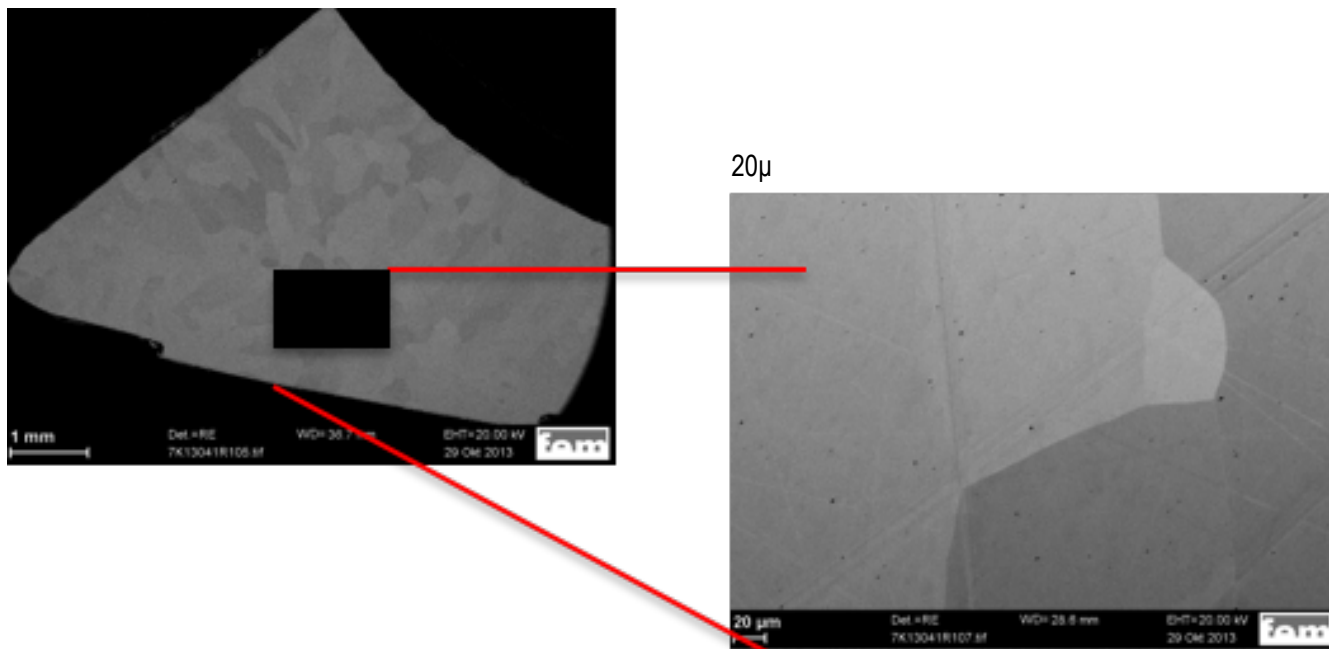• No significant change of microstructure during HIPing

Figure 11 - 95Pt5Co HIPed

RESULTS II: ALLOY DENSITY

Density of our test samples was measured using the Archimedes' Principle to determine levels of densification achieved through the HIP process. Although the density of our as-cast samples was already very high, HIP increased the levels to near 100%. This result is impressive and effectively puts castings on a par with wrought material. As can be seen in Table 3, HIP most effectively increased density in Pt95Ru5 and Pt90Rh10 and was slightly less effective for 90Pt10Ir, which we can see already starts out with higher as-cast density. This result correlates well with the generally lower levels of visible porosity seen in Pt90Ir10 cross sections from the 2011 study. Although 95Pt5Co was not tested for density, one would expect similar findings as in the case of 90Pt10Ir due to the lower levels of porosity in the as-cast state.

Table 3 Alloy Density Results

Alloy	Condition	Density [g/cm³]	Relative Density
95Pt5Ru	As-cast	20.32	98.4%
95Pt5Ru	HIPed	20.62	99.9%
90Pt10Ir	As-cast	21.39	99.5%
90Pt10Ir	HIPed	21.48	99.9%
90Pt10Rh	As-cast	19.58	98.2%
90Pt10Rh	HIPed	19.89	99.7%

Certain defect types either do not respond to HIP or have a lower densification response. A key limitation of the process is that only porosity that is fully subsurface will collapse; if pores are open to the surface of the casting in any way, they will not respond to HIP. This effect can be seen in the shrinkage porosity (Figure 12a). Another limitation of HIP is seen in gas pores. Pores created by gas are less responsive to HIP than shrinkage pores due to the pressure they contain. Rather than being eliminated, the pores are typically reduced in size by HIP as can be seen in the cross section below (Figure 12b).
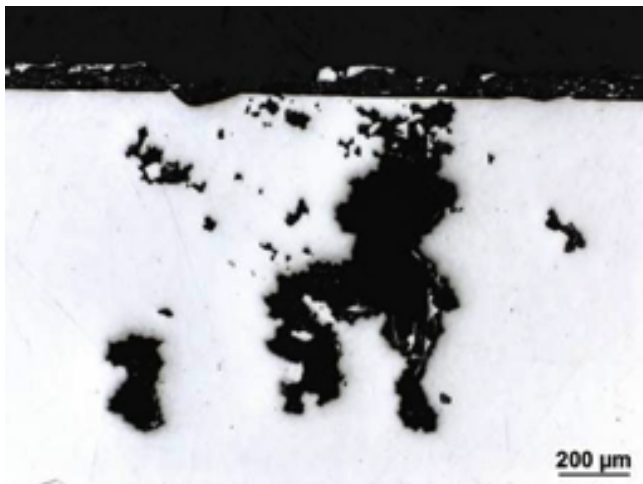

Figure 12a Surface connected shrinkage porosity in 95Pt5Ru

Figure 12b Sectioned Pt90Ir10 gas pore after HIP

RESULTS III: ALLOY HOMOGENEITY / EFFECTS ON SEGREGATION

RESULTS II: ALLOY DENSITY

Another aspect of our study was determining whether there had been any change in segregation of the alloying elements during high temperature heat treatment or HIP. By contrasting EDX mapping of 95Pt5Ru in the as-cast, heat treated, and HIPed conditions, we found that Ru segregated to the primary dendrites during solidification in a similar manner for all three conditions. Thus, neither heat treatment nor HIP changed the segregation of Ru. As can be seen in the comparative images in Figure 13, after HIP the dendrites have coarsened, but the microstructure is not negatively affected because the dendrites have arms that can coarsen without changing the overall size of the dendrite (Figure 13c)
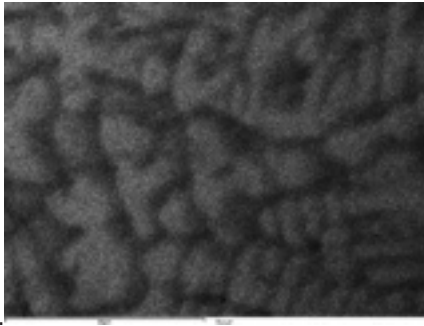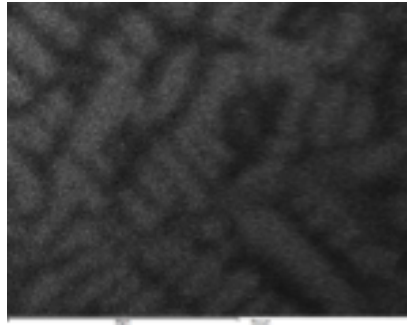
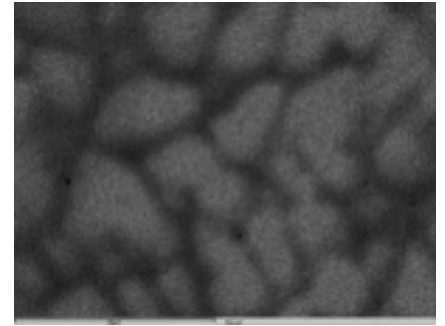Figure 13a Pt95Ru5 As-cast   Figure 13b Pt95Ru5 Heat   Figure 13c Pt95Ru5 HIPed
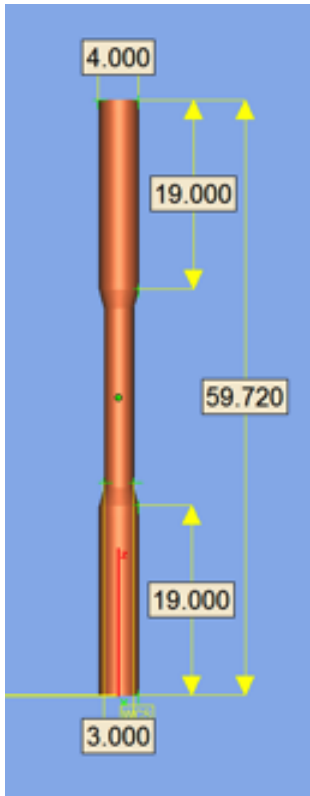
TENSILE TESTING

A literature search for mechanical properties data on cast platinum alloys showed few publications with the exception of micro-hardness values that are frequently cited by jewelry industry sources. One publication from 1978 by Ainsley and Rushforth was likely one of the earliest to look at tensile properties from actual castings versus the more commonly cited mill product values. These authors published values on nearly a dozen different platinum casting alloys including two that are also covered in our testing. It is notable that the values they published for the same alloys we tested were appreciably lower than ours in the as-cast state. Although it is not known why this was the case, a plausible explanation might be the difficulty of obtaining high quality cast test bars with the technologies available in 1978.

This relative scarcity of hard data is not so surprising given that sophisticated platinum casting is a relatively new development. It was not until the mid-nineties that induction machines capable of handling platinum's high temperature requirements became mainstream. Prior to that, small-scale oxyhydrogen torch melting was the only method available and inconsistent quality coupled with low pour weight capacity prevented investment casting from becoming a mainstream industrial process for platinum. All of that has of course changed and platinum based alloys are now routinely investment cast with induction melting methods on a global basis. While as-cast tensile properties of platinum alloys are of keen interest in their own right, we had additional motivation to perform this testing as a means to compare the strength characteristics of as-cast versus HIPed platinum alloys. In theory, the higher density of HIPed product would show increased values for a number of tensile properties, something we hoped to be able to prove. Table 4 outlines the testing plan that we followed to produce our data, followed by Figure 14 depicting the test bar geometry used for our tensile tests.

Table 4 Casting Specifications for Tensile Bars

Alloy	Condition	Density [g/cm³]	Test Bar Qty	Casting Condition
95Pt5Ru	1870	850	12	6 as-cast; 6 HIPed
90Pt10Ir	1870	850	8	4 as-cast; 4 HIPed
90Pt10Rh	1960	850	8	4 as-cast; 4 HIPed
95Pt5Co	1850	850	8	4 as-cast; 4 HIPed

1)   Total Bars: 36 ; minimum tests required: 18
2)   Test bar locations in "upper" and "lower" centrifuge orientation
3)   All waxes turned on lathe for dimensional precision
4)   All bars identically wax assembled with double end gates
5)   All bars cooled identically
5)   All bars HIPed to the same parameters

--Simple Design

--Heavy molten feed to gauge area with double end gates

--Directional solidification from interior of bar towards outer heavy sections

(Units in mm)

Before

Figure 14 Test bar geometry

Before launching our tensile tests we thought it prudent to compare the porosity levels found in our as-cast test bar geometry versus those from the test design used in the porosity analysis reported in the 2011 research. In Figure 15a is a 50X photomicrograph of a cross-section taken from the gauge area of a 95Pt5Ru test bar. In comparing this with Figure 15b, a 95Pt5Ru cross section at 50X derived from our earlier research, we note a somewhat different distribution in the porosity. The test bar has a centerline orientation to the microshrinkage, whereas the ring geometry has more scattered microshrinkage. Notwithstanding the notable differences seen in the samples below, the important point here is that both of these geometries produce what can be considered a near-optimal condition for 95Pt5Ru in the as-cast condition. Conversely, if we refer to Figure 16 depicting a typical jewelry design, we can easily surmise that solidification of this geometry will be more challenging due to the thick-thin orientation and lack of ability to directionally solidify. Thus, tensile results reflect an optimal condition in comparison with much of the real world cast product.
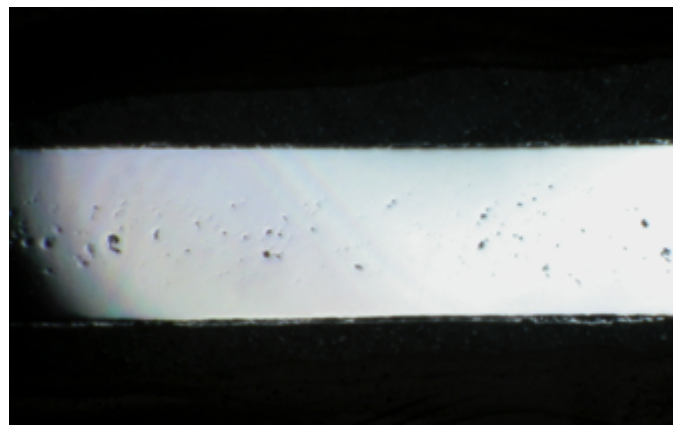

Figure 15a 95Pt5Ru cross-section of test bar gauge area at 50X.
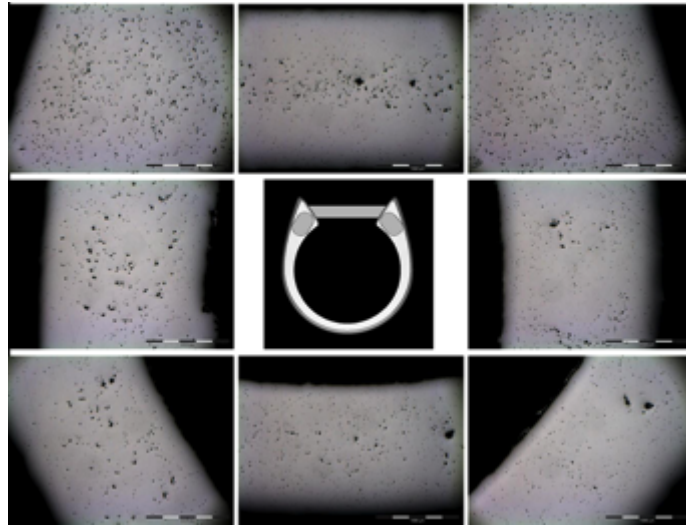Porosity is concentrated at centerline

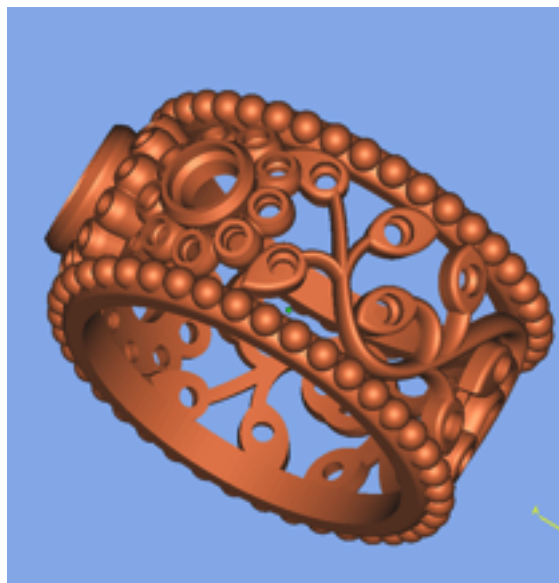Figure 15b 95Pt5Ru cross-section of double sprued test geometry. Micro-porosity is scattered more uniformly

Figure 16 Typical jewelry design: thick to thin junctions, minimal areas to attach feed sprues,
no possibility for directional solidification

RESULTS IV: TENSILE TESTING

The results in Table 5 report values for Yield Strength (YS), Ultimate Tensile Strength (UTS), Elongation (ε) and Reduction of Area (ROA). Yield strength describes stress levels above which plastic deformation occurs and will generally increase with decreasing grain size. Following yielding, the material work hardens by the generation of dislocations. As a consequence, the required stress for further deformation increases until the ultimate tensile strength is reached. In metals, the UTS values will generally correlate with Vickers hardness values. Figure 17 shows the tensile results in graphic format.

Table 5 Tensile Properties (AC = as-cast ; HIP = hot isostatic pressed)

| Alloy Composition & Condition	YS [MPa]	UTS [MPa]	ε [%]	ROA [%]	ROA % Change
95Pt5Ru - AC	225	412	30	55	---
95Pt5Ru - HIP	236	420	39	87	+ 32
90Pt10Ir - AC	219	353	33	90	---
90Pt10Ir - HIP	226	358	36	87	-3
90Pt10Rh - AC	140	330	37	64	---
90Pt10Rh - HIP	144	333	43	89	+25
95Pt5Co - AC	220	452	36	76	---
95Pt5Co - HIP	189	449	38	82	+6

Figure 17 Tensile Results
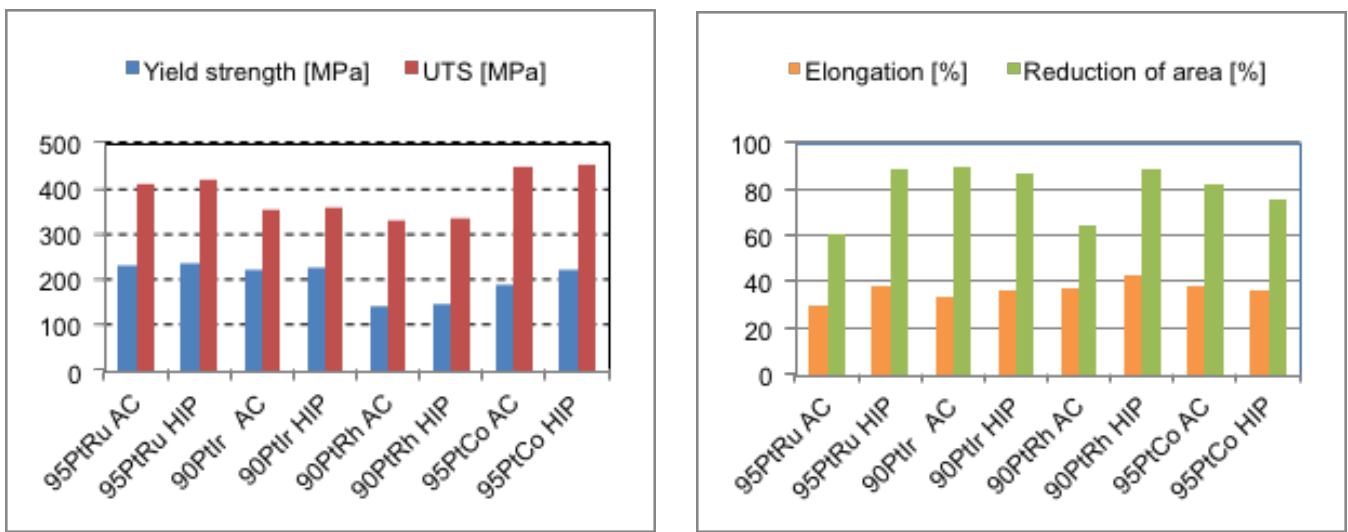
At strains above UTS, which marks the maximum of the stress-strain curve (Figure17), the cross-section is locally reduced through necking of the sample. Further deformation is localized in the necking region and as a consequence the required stress for further deformation is continually decreasing until failure occurs. The total elongation (ε) indicates how much plastic deformation the material can withstand. Pores in the material will significantly reduce the elongation because they act as stress concentration sites. The effect of pores is even more pronounced on the reduction of area, which indicates how much necking occurs until the sample finally fails. While UTS or hardness are clearly important properties to measure, they are not necessarily the most critical properties to predict failure in a broad number of applications. When it comes to fatigue life, elongation and reduction of area are generally viewed as being more important. Specifically, in cases where subsequent cold working of the material is involved, an increased ability to bend before cracking is of paramount importance.

The HIP treatment affects the mechanical properties of the four alloys in a different way. For all alloys the effect on YS and UTS is rather low. For 90Pt10Ir and 95Pt5Co there is little effect on elongation and ROA. However, for 95Pt5Ru and 90Pt10Rh a significant increase in elongation and ROA is found through HIPing. These results correlate well with the porosity levels of the different alloys, and are a clear indication that reduction of porosity increases ductility in the alloys.

Another interesting observation came from our analysis of UTS scatter in the sample population. The graphs below demonstrate the difference in spread between the as-cast and HIPed groups (Figure 17). The HIPed bars exhibit a very tight distribution, whereas the as-cast bars are more scattered. This result correlates well with our observations of lower porosity levels together with a more homogeneous grain size and structure in the HIPed samples.
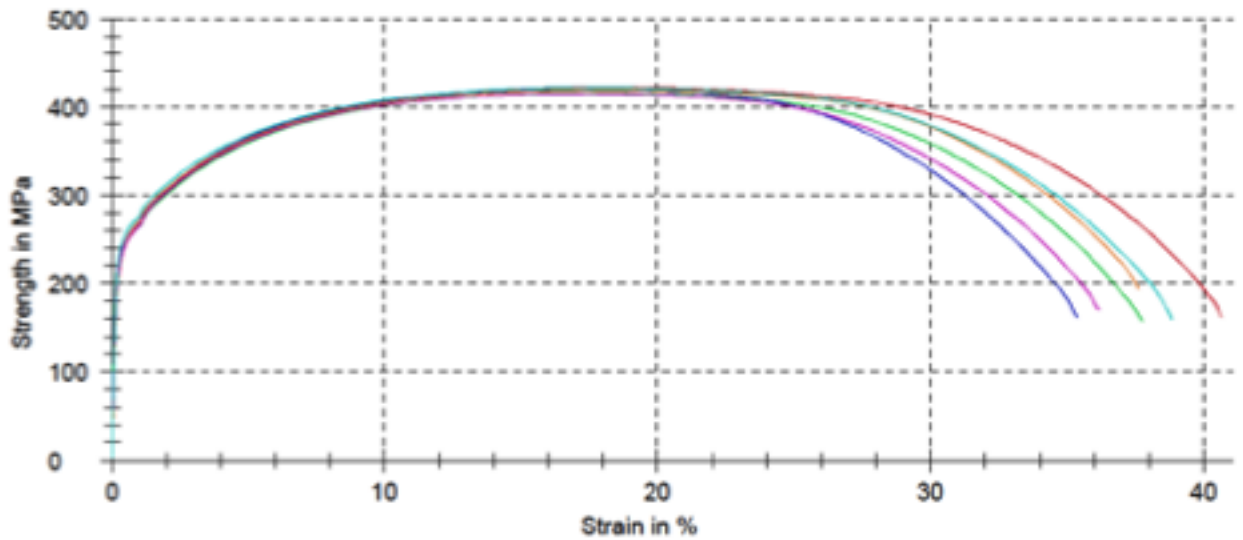
As-cast
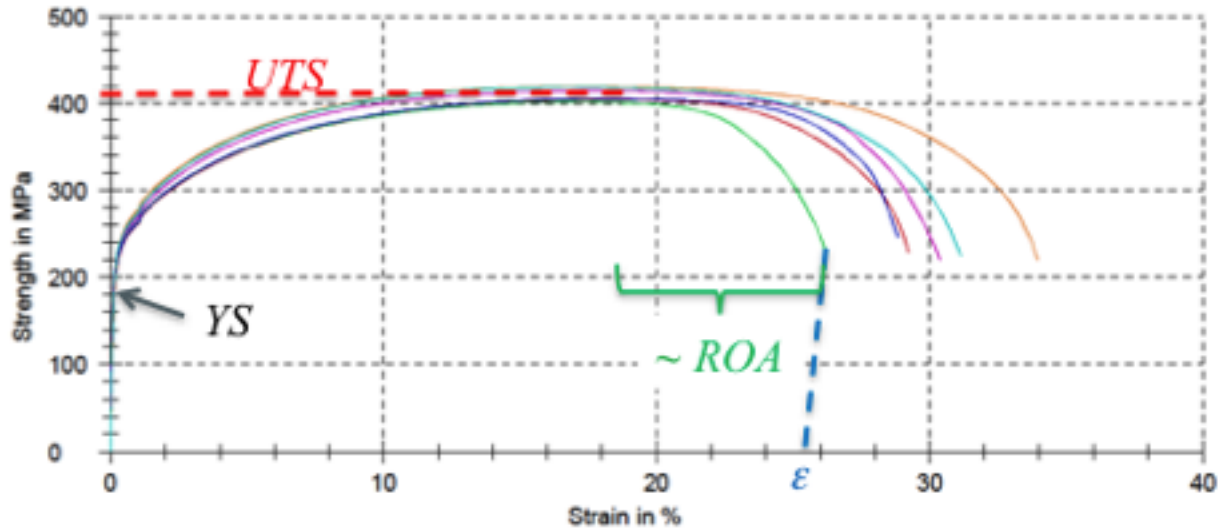
HIPed

Figure 18 UTS scatter of 95Pt5Ru

As stated above, reduction of area values posted the most impressive gains in the HIPed product. This property is of particular interest in the jewelry industry given the substantial amount of bending and forming that is inherent in stone setting, engraving, sizing, and myriad bench operations. Reduction of area indicates a material's ductility and is crucial to successful performance in many of these operations. Figure 18a and 18b demonstrate a profound visual difference in ductility between the test bar fractures in the as-cast and HIPed 95Pt5Ru.
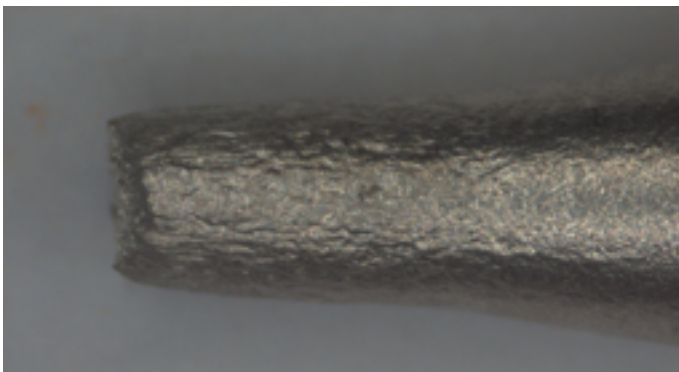

Figure 19a 95Pt5Ru As-cast 55% ROA

Figure 19b 95Pt5Ru HIPed 87% ROA RESULTS V: MICRO-

HARDNESS TESTING

Hardness is a very important property for many platinum alloys due to frequent use in jewelry and other applications where a high resistance to surface wear is desired. Due to the relative softness of platinum alloys, micro-hardness testing is generally the method of choice. An interesting aspect of micro-hardness testing that bears closer examination is the significant difference in reported values depending upon the load used in the test. According to the philosophies of the individual lab doing the work, typical loads used are either 100, 500, or 1000 grams. Figure 19 is a chart demonstrating the differences in Hv values and indents by load on a single sample of 95Pt5Ru.
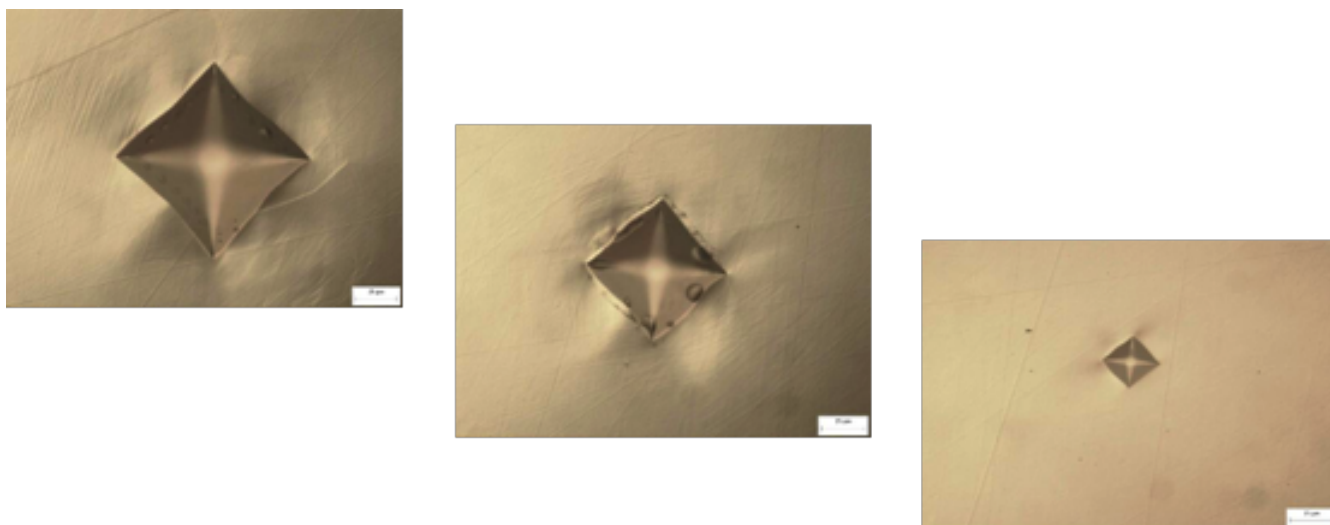

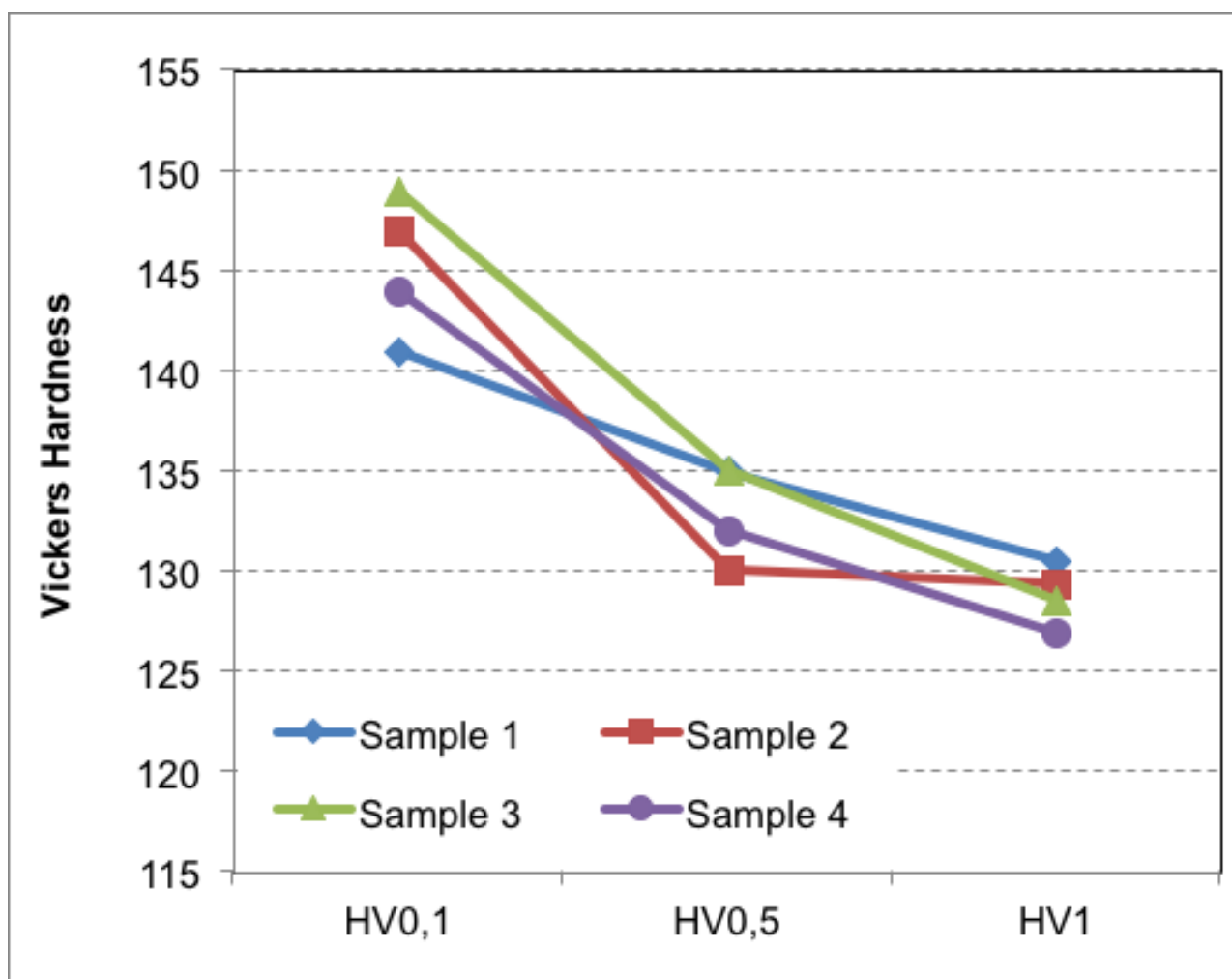Figure 20 HV indents on 95Pt5Ru at 1000/500/100g loads

Figure 21 Micro-hardness of 95Pt5Ru results by load. Hardness decreases with increasing load

With increasing loads the Vickers hardness number and the scatter between the individual measurements decreases. At loads of 1000 grams or above the values become constant. In the case of lower loads, this may be at the expense of the accuracy of the result for several reasons. With lower loads, the hardness indent becomes smaller and the hardness information originates from a smaller sample volume. Therefore, pores, inclusions or other material inhomogeneities affect the obtained results strongly due to their relative concentration in the indent itself. Conversely, if there are pores outside of the indent and the indent itself was too small to capture these, the sample would yield a higher result than the bulk material would warrant. The measurement principle of Vickers hardness testing requires measuring both diagonals of the hardness indent. With small indents the accuracy of such measurements becomes lower and thereby the scatter among the individual hardness measurements increases. Therefore, 1000 grams would appear to be the more suitable load for testing of macroscopic platinum alloy samples. Detailed recommendations and standardizations of Vickers hardness testing can be found in national and international standards, such as DIN EN ISO 6507-1:2006-03. Having said that, it must be acknowledged that the jewelry industry in North America is not standardized in its approach to measuring and reporting Vickers hardness. For example, the Hv values in this paper will appear very low in comparison to the typical reported hardness levels for these well-known alloys simply because of the high load we chose to apply. Further work should be pursued towards the use of a more standardized approach with respect to micro-hardness testing and reporting across the jewelry industry. This would improve understanding of material performance expectations by designers and manufacturers and help predict wear resistance in a more standardized manner.

Table 6 reports Vickers hardness values for three of the tested alloys. With the possible exception of 950 PtRu, all alloys report values that are so close in the before and after HIP conditions that any difference is seen as essentially inconsequential. Even the 950 PtRu that shows a 12-point spread is not considered enough to be characterized as an appreciably harder material by performance. Thus we can conclude that hardness is not significantly impacted by HIP on the platinum-based alloys we tested.

Table 6 Vickers Micro-hardness Results

Vickers Hardness HV1			
Alloy	As-Cast	HIPed	Heat Treated
90Pt10Ir	113	111	123
90Pt10Rh	89	89	n/a
95Pt10Ru	113	125	128
95Pt5Co	126	122	N/A

CONCLUSIONS

The most significant impact of hot isostatic pressing on platinum-based alloys is a reduction in porosity. Reduced levels of porosity have several associated benefits, including a marked increase in ductility in the majority of the alloys tested without sacrificing strength. Of the tensile properties tested, the most impressive response was found in the values for ROA, a key indicator of an alloy's ductility.

Another meaningful result demonstrated through our research was HIP's effect on grain structure and size. Although further testing is needed to fully characterize this aspect, our initial results appear to have a finer and more uniform grain size and structure in the HIPed samples, at least for the alloy 95Pt5Ru.

Findings also confirm that the response to HIP is strongly impacted by the alloy's composition. 95Pt5Ru benefits the most due to its higher levels of porosity present in the as-cast condition, and 95Pt5Co having lower porosity benefits the least.

Further work is recommended to assess the impact of qualitative changes in the HIPed product on manufacturing operations. Empirical evidence strongly suggests greater ease in bench operations due to the elimination of sub-surface micro-porosity and a generally more consistent metallurgical condition, including uniformity of grains. Polishing, engraving, and setting can all benefit from a less porous and more consistent microstructure. In addition, the increased ductility should, in theory, result in a lower number of failures during metal bending and forming operations.

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