# *Microstructure Analysis of Selected Platinum Alloys*

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*Metallographic analysis can be used to determine the microstructure of platinum alloys in order to set up working cycles and to perform failure analyses. A range of platinum alloys used in jewellery and industrial applications was studied, including several commonly used jewellery alloys. Electrochemical etching was used to prepare samples for analysis using optical metallography and additional data could be obtained by scanning electron microscopy and energy dispersive spectroscopy. The crystallisation behaviour of as-cast alloy samples and the changes in microstructure after work hardening and annealing are described for the selected alloys.*

## Introduction

Optical metallography is a widely used investigation technique in materials science. It can be used to describe the microstructure of a metal alloy both qualitatively and quantitatively. Here, the term 'microstructure' refers to the internal structure of the alloy as a result of its composing atomic elements and their three-dimensional arrangement over distances ranging from 1 micron to 1 millimetre.

Many alloy properties depend on the microstructure, including mechanical strength, hardness, corrosion resistance and mechanical workability. Metallography is therefore a fundamental tool to support research and failure analysis (1–3). This is true for all industrial fields where alloys are used. A great deal of literature is available on the typical methods used in optical metallography (4–6).

A large amount of useful information is available in the literature for precious metals in general (7–10). However, there is less information specifically focussed on platinum and its alloys.

The present work aims to give some examples of platinum alloy microstructures, both in the as-cast and work hardened and annealed conditions, and to demonstrate the usefulness of optical metallography in describing them. This paper is a revised and updated account of work that was presented at the

 

24th Santa Fe Symposium® on Jewelry Manufacturing Technology in 2010 (11).

## Materials and Methods

A wide variety of platinum alloys are used in jewellery (12–18) and industrial applications (10, 19–21). Different jewellery alloys are used in different markets around the world, depending on the specific country's standards for precious metal hallmarking. The alloys whose microstructures are discussed here are listed in Table I. These do not represent all the alloys available on the market, but were chosen as a representative sample of the type of results that can be obtained using metallographic techniques. The related Vickers microhardness of each alloy sample, measured on the metallographic specimen with a load of 200 gf (~2 N) in most cases, is given for each microstructure.

If metallographic analysis is aimed at comparing the microstructure of different alloys in their as-cast condition, the initial samples must have the same size and shape. Mould casting or investment casting can produce different microstructures, with different grain sizes and shapes, depending on parameters such as mould shape, size and temperature, the chemical composition of the mould, etc. Therefore, whenever possible, the specimens for the present study were prepared under conditions which were as similar as possible, including the casting process. The specimens were prepared by arc melting and pressure casting under an argon atmosphere to the shape shown in Figure 1. A Yasui & Co. Platinum Investment was used, with a final flask preheating temperature of 650ºC. The captions of the micrographs specify whether the original specimen is of the type described above.

The preparation of the metallographic specimens consists of the following four steps: sectioning, embedding the sample in resin, polishing the metallographic section, and sample etching for microstructure

Table I

Selected Platinum Alloys

| Composition, wt% | Melting range[a], ºC | Vickers microhardness[b], $HV_{200}$ |
|---|---|---|
| Pt | 1769 | 65[c] |
| Pt-5Cu[d] | 1725–1745 | 130 |
| Pt-5Co[d] | 1750–1765 | 130 |
| Pt-5Au[d] | 1740–1770 | 127 |
| Pt-5Ir[d] | 1780–1790 | 95 |
| Pt-5Ru[d] | 1780–1795 | 125 |
| 70Pt-29.8Ir[e] | 1870–1910 | 330 |
| 70Pt-30Rh | 1910[f] | 127 |
| 90Pt-10Rh | 1830–1850[f] | 95 |
| 60Pt-25Ir-15Rh | n/a | 212 |

[a]*Some melting ranges are not given as they have not yet been reported*

[b]*The microhardness value refers to the microstructure of samples measured in this study and reported in the captions of the Figures*

[c]$HV_{100}$

[d]*These alloys are among the most common for jewellery applications. Where it is not specified, it is assumed that the balance of the alloy is platinum*

[e]*This alloy composition is proprietary to 8853 SpA, Italy*

[f]*Solidus temperature*

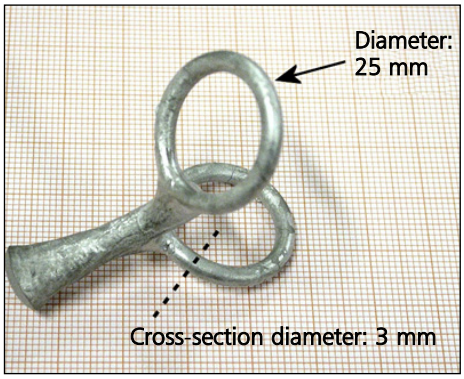


*Fig. 1. General shape of specimens prepared by investment casting for this study. The microstructures of different alloys obtained by investment casting can be compared, provided that the specimens have the same size and shape. The dashed line shows the position of the metallographic sections examined in these samples*

detection. The detailed description of these steps will not be given here, as they have been discussed in other works (4–10).

Further advice relevant to platinum alloys was given in the 2010 Santa Fe Symposium paper (11) and in this Journal (22). In these papers, procedures for the metallographic analysis of most platinum alloys are described. The samples for the present study were prepared by electrolytic etching in a saturated solution of sodium chloride in concentrated hydrochloric acid (37%) using an AC power supply, as described previously (22).

## Microstructures of the Platinum Alloys

In this section the microstructures of the selected platinum alloys in different metallurgical conditions are presented. As already stated, this selection is a representative sample and not a complete set of the platinum alloys which are currently on the market.

### As-Cast Microstructures: Metallography of Crystallisation

Examination of the as-cast microstructures shows the variation in size and shape of the grains in different platinum alloys. However, a noticeable dendritic grain structure is quite common. The largest grain size was found in platinum with 5 wt% copper (Pt-5Cu) (Figure 2) and platinum with 5 wt% gold (Pt-5Au) (Figure 3), with sizes up to 1 mm and 2 mm, respectively. The Pt-5Au alloy sample also shows shrinkage porosity between the dendrites. The core of the dendritic grains showed a higher concentration of the element whose melting temperature was the highest in both cases. This behaviour, known as 'microsegregation', has been widely described (12, 23, 24). Electrolytic etching tended to preferentially dissolve the interdendritic copper- or gold-rich regions, respectively. In a platinum with 5 wt% iridium (Pt-5Ir) alloy (Figure 4), since iridium has the higher melting temperature, the dendritic crystals were enriched in iridium in the first solidification stage.

It is important to point out that the higher or lower visibility of microsegregation within the dendrites is not directly related to the chemical inhomogeneity, but to the effectiveness of the electrolytic etching in

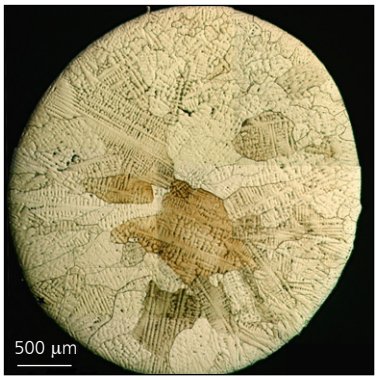


*Fig. 2. As-cast Pt-5Cu alloy showing dendritic grains with copper microsegregation (sample shape as in Figure 1; flask temperature 650°C; microhardness 130 ± 4 $HV_{200}$)*

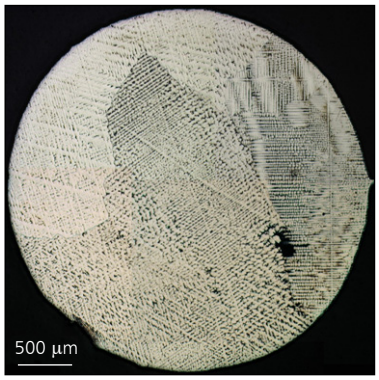


*Fig. 3. As-cast Pt-5Au alloy showing shrinkage porosity between the dendrites (sample shape as in Figure 1; flask temperature 650°C; microhardness 127 ± 9 $HV_{200}$)*

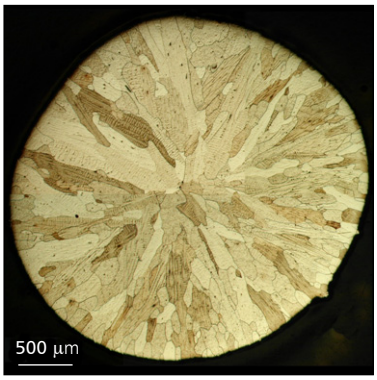


*Fig. 4. As-cast Pt-5Ir alloy with columnar grains (sample shape as in Figure 1; flask temperature 650°C; microhardness 95 ± 2 $HV_{200}$)*

 

revealing it. For example, the microsegregation in the platinum with 5 wt% cobalt (Pt-5Co) alloy is hardly visible in Figure 5, despite being easily measurable by other techniques (24).

Scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) are very effective in showing the presence of microsegregation. Figure 6 shows an as-cast sample of a platinum with 25 wt% iridium and 15 wt% rhodium alloy (60Pt-25Ir-15Rh). The SEM backscattered electron image is shown in Figure 7. The EDS maps in Figures 8–10 give the elemental distribution on the etched surface. If the maps were obtained on the polished surface the approximate concentration of each element may be different due to the etching process and a possible preferential dissolution of different phases of the alloy.

However, because EDS is a semi-quantitative method, it can only give the general distribution of the elements on the metallographic section. It is worthwhile remembering that metallographic preparation reveals only a few microstructural features. By changing the preparation or the observation technique, some microstructural details may appear or become more clearly defined, while others remain invisible.

The melting range of the alloy and the flask preheating temperature affect the size and shape of grains significantly. In order to decrease the dendritic size and obtain a more homogeneous microstructure, the temperature of the material containing the solidifying alloy is lowered as much as possible. The effectiveness of such an operation is, however, limited by

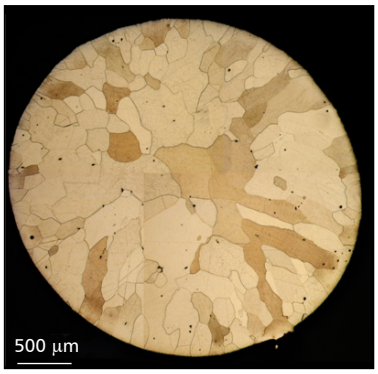


*Fig. 5. As-cast Pt-5Co alloy with small gas porosity (sample shape as in Figure 1; flask temperature 650°C; microhardness 130 ± 6 $HV_{200}$)*

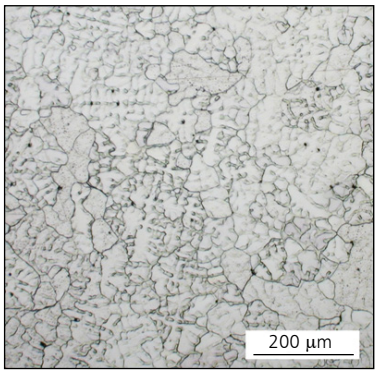


*Fig. 6. 60Pt-25Ir-15Rh alloy cast in a copper mould. From the transverse section of an ingot (microhardness 212 ± 9 $HV_{200}$)*

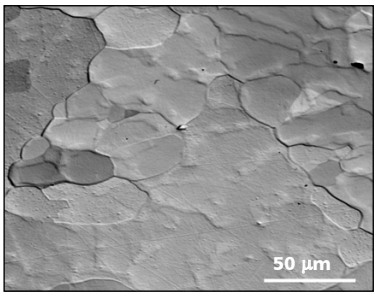


*Fig. 7. 60Pt-25Ir-15Rh alloy: scanning electron microscopy (SEM) backscattered electron image of the etched sample. The sample is the same as that shown in Figure 6*

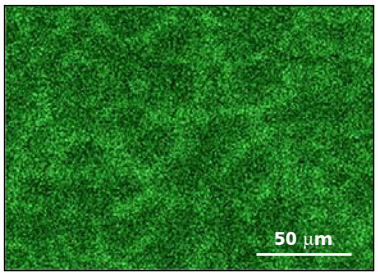


*Fig. 8. 60Pt-25Ir-15Rh alloy: energy dispersive spectroscopy (EDS) platinum map acquired on the surface seen in Figure 7, showing the platinum microsegregation. The network of high platinum content shows this approximate composition (wt%): 72Pt-14Ir-14Rh*

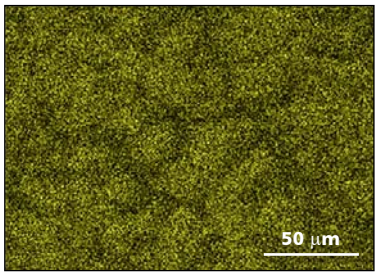


*Fig. 9. 60Pt-25Ir-15Rh alloy: energy dispersive spectroscopy (EDS) iridium map acquired on the surface seen in Figure 7. The iridium concentration is lower where that of platinum is higher*

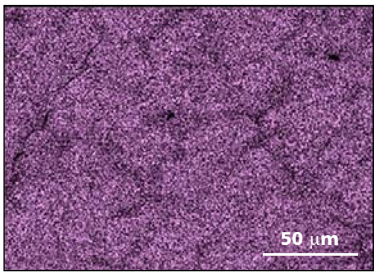


*Fig. 10. 60Pt-25Ir-15Rh alloy: energy dispersive spectroscopy (EDS) rhodium map acquired on the surface seen in Figure 7. The rhodium distribution follows the behaviour of iridium. The zones of higher iridium and rhodium content show this approximate composition (wt%): 55Pt-28Ir-17Rh*

the melting range and by the chemical composition of the alloy. An example of the flask temperature effect is shown in Figure 11 for Pt-5Ir poured into a flask with a final preheating temperature of 890ºC. This microstructure is to be compared with that in Figure 4, in which a flask preheating temperature of 650ºC was used.

A smaller grain size was observed in the platinum with 5 wt% ruthenium (Pt-5Ru) alloy, which showed a more equiaxed grain (Figure 12) with a grain size of about 200 μm. The addition of ruthenium led to finer grains in the platinum alloy.

Pouring the alloy in a copper mould produces a smaller grain size due to the high cooling rate, as visible in Figure 6 and Figures 13–15. In this case, the high iridium and rhodium content also contributed to the lower grain size in the as-cast sample.

Homogenising thermal treatments result in a microstructural change. Comparing Figure 16 with Figure 17 highlights a reduction in microsegregation in Pt-5Cu as a consequence of a homogenisation treatment performed at 1000ºC for 21 hours.

## Work Hardened and Annealed Microstructures: Metallography of Deformation and Recrystallisation

Optical metallography can reveal the changes in microstructure that occur after work hardening and recrystallisation thermal treatments and allows recrystallisation diagrams like the one in Figure 18 to

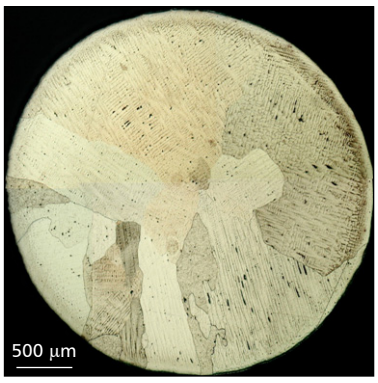


*Fig. 11. As-cast Pt-5Ir alloy (sample shape as in Figure 1; flask temperature 890ºC; microhardness 105 ± 2 $HV_{200}$)*

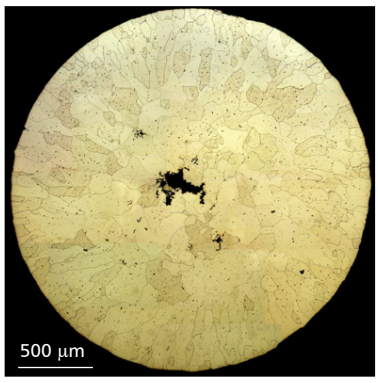


*Fig. 12. As-cast Pt-5Ru alloy showing shrinkage porosity at the centre of the section (sample shape as in Figure 1; flask temperature 650ºC; microhardness 125 ± 5 $HV_{200}$)*

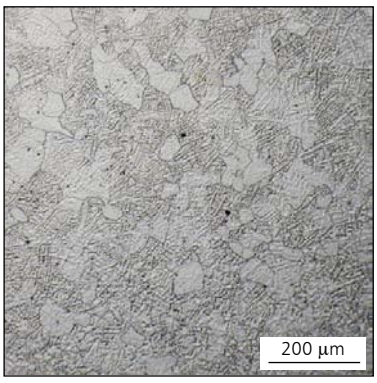


*Fig. 13. 70Pt-29.8Ir alloy: cast in a copper mould. From an ingot transverse section. A high iridium content contributes to grain refinement (microhardness 330 ± 4 $HV_{200}$)*

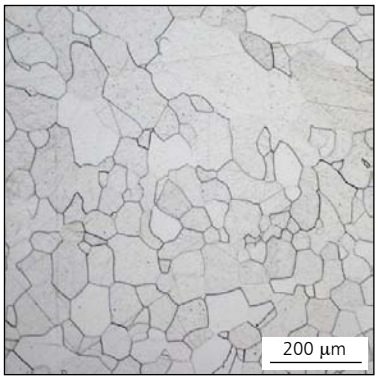


*Fig. 14. 70Pt-30Rh alloy: cast in a copper mould. From the transverse section of an ingot. A high rhodium content enhances the grain refinement (microhardness 127 ± 9 $HV_{200}$)*

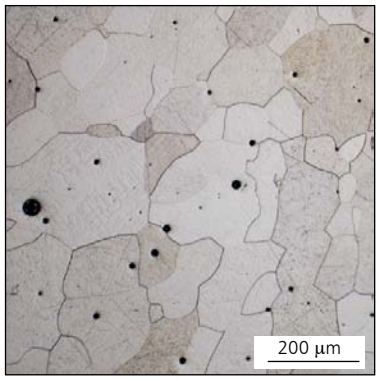


*Fig. 15. 90Pt-10Rh alloy: cast in a copper mould. From the transverse section of an ingot. The gas porosity is visible (microhardness 95 ± 5 $HV_{200}$)*

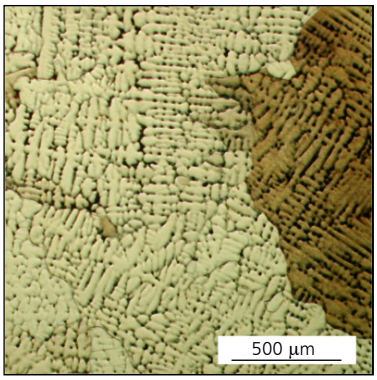


*Fig. 16. Higher-magnification image of as-cast Pt-5Cu alloy showing dendritic grains with copper microsegregation (microhardness 130 ± 4 $HV_{200}$). Compare with Figure 17*

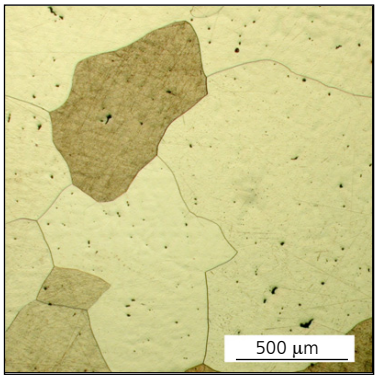


*Fig. 17. Microstructure of Pt-5Cu alloy after thermal treatment at 1000°C for 21 hours. The micro-segregation of copper is reduced (micro-hardness 120 ± 4 $HV_{200}$). Compare with Figure 16*

be drawn. This makes it a valuable aid in setting up working cycles. It is necessary to establish the right combination of plastic deformation and annealing treatment in order to restore the material's workability. This allows suitable final properties to be achieved.

An example of the changes in microstructure after various stages of work hardening and annealing is shown in Figure 19 for the 60Pt-25Ir-15Rh alloy. This can be compared to the as-cast structure shown in Figure 6.

Drawn wires show a very different microstructure along the drawing (longitudinal) direction in comparison to the transverse direction (Figures 20–22 for Pt-5Au). However, after annealing, the microstructure becomes homogeneous and the fibres formed after to the drawing procedure are replaced by a recrystallised microstructure (Figures 23 and 24). Using the techniques described elsewhere (11), analyses can be performed even on very thin wires, as shown in Figure 25 for a platinum 99.99% wire of 0.35 mm diameter.

It is worth pointing out that some binary platinum alloys have a miscibility gap at low temperatures, as shown by their phase diagrams (19, 20, 25). Examples of this are given in Figures 26 and 27 for Pt-Ir and Pt-Au, respectively. Similar behaviour is observed for Pt-Co, Pt-Cu and Pt-Rh alloys.

As a consequence, a biphasic structure is expected of each of them. However, this may not occur for various reasons. The phase diagrams refer to equilibrium conditions, which hardly ever correspond to the as-cast conditions. One of the two phases is sometimes present but in low volumetric fraction, due to the chemical composition of the alloy, in which one of the two elements has a low concentration. Furthermore, the thermal treatments may have homogenised the alloy. Finally, the metallographic preparation may not be able to reveal such biphasic structures. Therefore, it is necessary to use other analytical techniques to detect the type and concentration of the alloy phases. Only in specific cases can the biphasic structure be revealed.

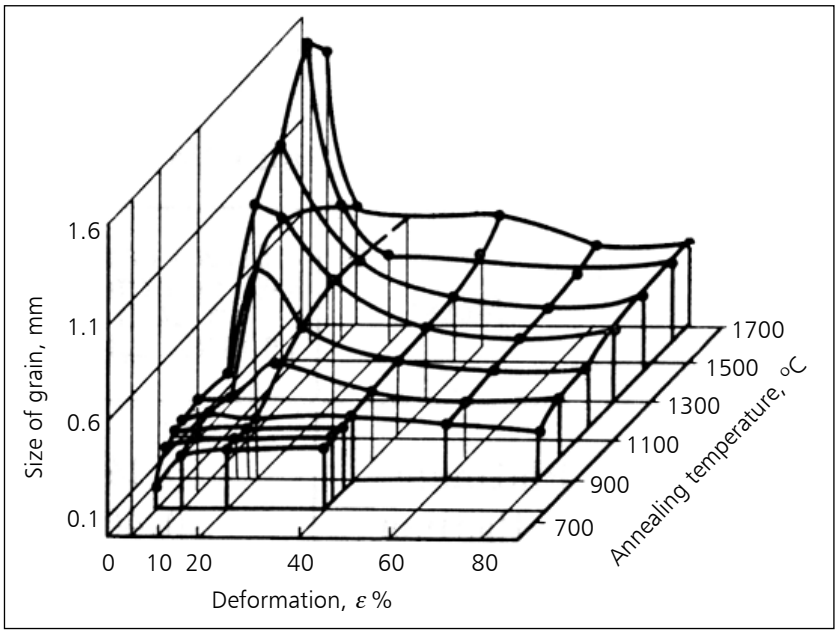


*Fig. 18. Recrystallisation diagram of a platinum-rhodium alloy annealed at a set temperature for a given time after a deformation of ε %. Adapted from (10). By increasing the annealing temperature the grain size increases. During annealing the grain size also increases if the previous deformation is reduced*

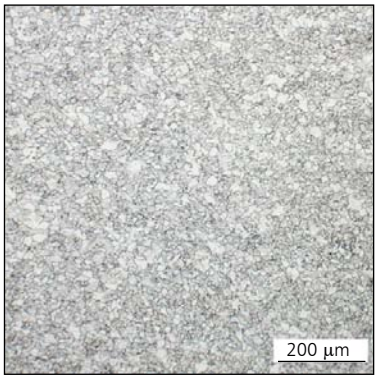


*Fig. 19. 60Pt-25Ir-15Rh alloy: from the transverse section of an ingot, after various stages of work hardening and annealing (microhardness 212 ± 5 $HV_{200}$). Compare with the as-cast sample shown in Figure 6*

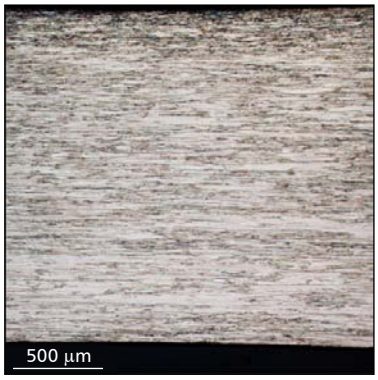


*Fig. 20. Pt-5Au alloy: longitudinal section (along the drawing direction) of a drawn cold worked wire (microhardness 190 ± 4 $HV_{200}$)*

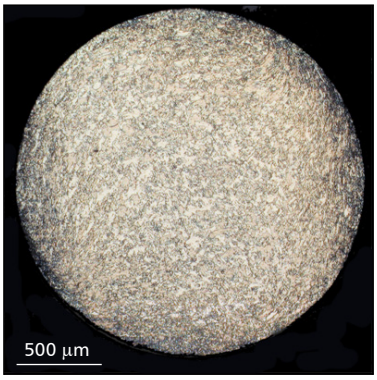


*Fig. 21. Pt-5Au alloy: transverse section of the drawn cold worked wire seen in Figure 20 (microhardness 190 ± 4 $HV_{200}$)*

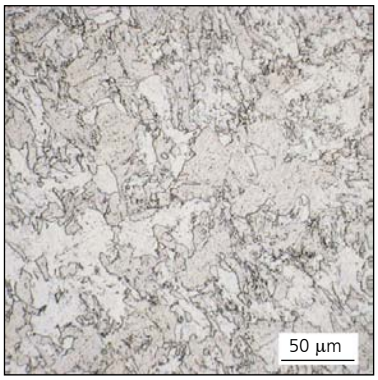


*Fig. 22. Pt-5Au alloy: detail of Figure 21 showing the deformation of the grains*

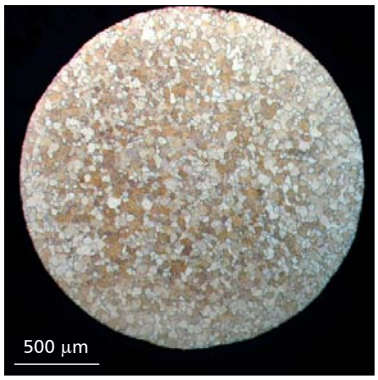


*Fig. 23. Pt-5Au alloy: transverse section of the wire seen in Figure 21, after oxygen-propane flame annealing (microhardness 104 ± 6 $HV_{200}$)*

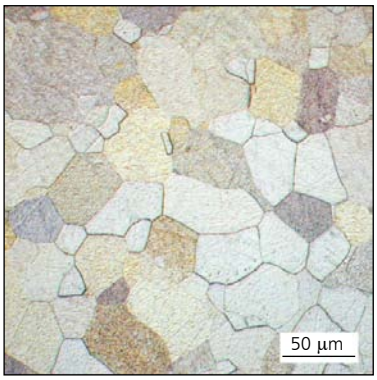


*Fig. 24. Pt-5Au alloy: detail of Figure 23, showing the recrystallised grains*

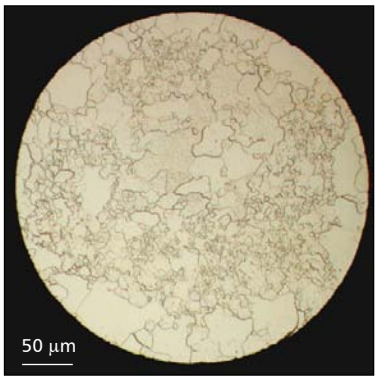


*Fig. 25. Pt 99.99% wire: transverse section of the wire after various stages of drawing and annealing (diameter 0.35 mm; microhardness 65 ± 3 $HV_{100}$)*

The best results in working platinum alloys are generally achieved by hot forging the ingot during the first stages of the procedure. Metallography shows the differences between a material that has been cold worked and annealed (Figures 28 and 29 for Pt-5Cu) and a material that has been hot forged (Figures 30 and 31). Hot forging more easily achieves a homogeneous and grain-refined microstructure, free of defects. This is due to the dynamic recrystallisation that occurs during hot forging (26).

## The Limits of Metallography

Optical metallography is only the first step towards the study of the microstructure of an alloy. A wide variety of analytical techniques can be used alongside

 

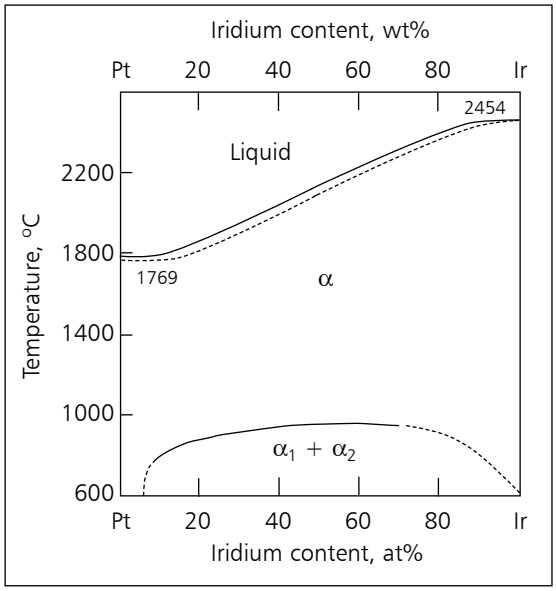


*Fig. 26. Pt-Ir phase diagram showing a miscibility gap at low temperatures (20)*

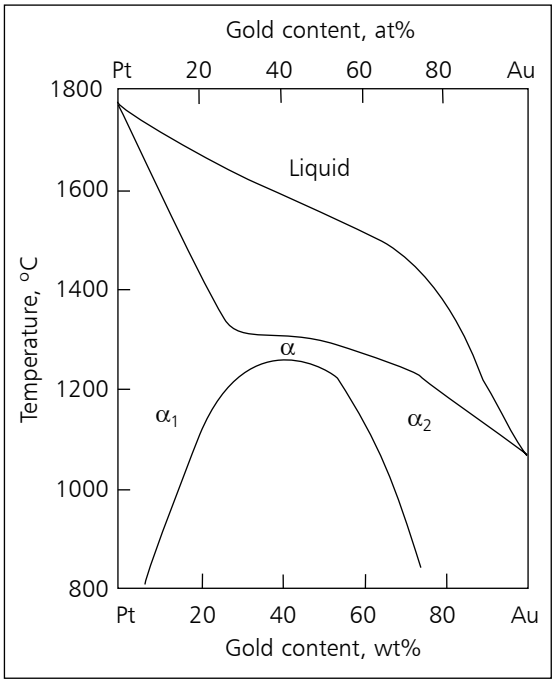


*Fig. 27. Pt-Au phase diagram showing a miscibility gap at low temperature (25)*

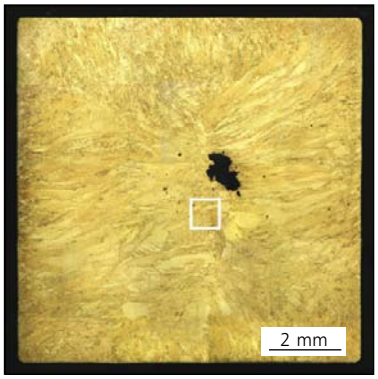


*Fig. 28. Pt-5Cu alloy: from a transverse section of a 19 mm × 19 mm ingot, which was rod milled, annealed in a furnace and finished at 10 mm × 10 mm by drawing. The sample shows residual coarse grain microstructure from the as-cast condition and fractures along the bar axis (microhardness 208 ± 13 $HV_{200}$). The small square shows the position of the detail seen in Figure 29*

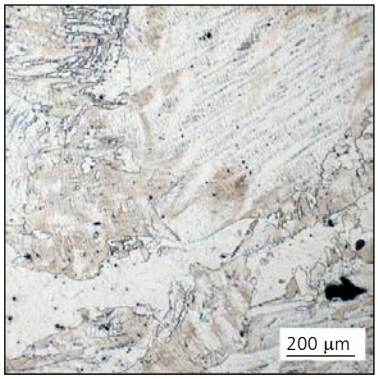


*Fig. 29. Pt-5Cu alloy: detail of Figure 28, with coarse grains and small opened cracks evident*

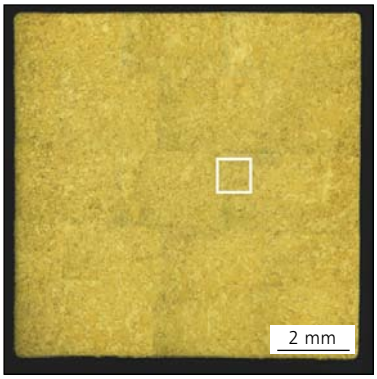


*Fig. 30. Pt-5Cu alloy: from a transverse section of a 19 mm × 19 mm bar, which was hot hammered, torch annealed and finished by drawing. The sample has homogeneous microstructure with small grain size (microhardness 200 ± 9 $HV_{200}$). The small square shows the position of the detail seen in Figure 31*

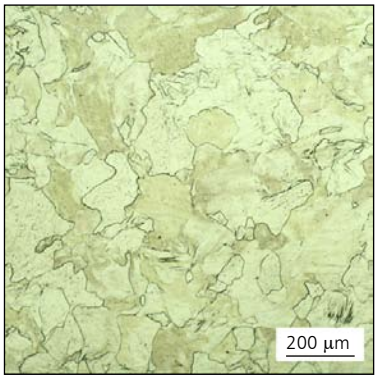


*Fig. 31. Pt-5Cu alloy: detail of Figure 30, showing recrystallised grains partially deformed due to the work hardening*

it to provide a far more complete knowledge of the microstructure. One of the most widely used techniques is SEM. In addition to this, EDS allows the relative concentration of the contained chemical elements to be determined, as shown in Figures 8–10. Further studies can be performed by X-ray diffraction (XRD), which reveals the different crystal phases present in the alloy.

When working with platinum alloys, often only very small specimens are available, therefore more recent techniques may be required in order to study them. One of these is the focused ion beam (FIB) technique, which can produce microsections of a specimen (27, 28). The microsections are then analysed by other techniques, such as transmission electron microscopy (TEM). In this case the details of microstructure can be detected due to the high spatial resolution of the technique. The crystal structure of the primary and secondary phases can be studied by electron diffraction. Another interesting technique is nano-indentation, performed with micron-sized indenters, which allows hardness measurements to be performed with a spatial resolution far better than that attainable with ordinary micro-indenters. The data obtained from these measurements allows the measurement of fundamental mechanical properties of the alloy, such as the elastic modulus (Young's modulus) (29).

## Conclusions

The metallographic analysis of platinum alloys can be profitably carried out by using a specimen preparation methodology based on the techniques used for gold-based alloys. However, electrochemical etching is required in order to reveal the alloy microstructure and observe it by optical microscopy. A saturated solution of sodium chloride in concentrated hydrochloric acid can be successfully used for a great many platinum alloys, both in the as-cast condition and after work hardening. Optical metallography provides essential data on the alloy microstructure which can be used in setting up the working procedures. Other techniques can be used alongside it to achieve a more complete knowledge of the material, the effects of the working cycles on it, and to interpret and explain any remaining problems.

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