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Paolo Battaini holds a degree in nuclear engineering, with a bias towards materials. He specialises in electron microscope techniques and is an expert in failure analysis applied to various industrial sectors and in metallurgy of precious metals. Since 1984 he has focused on precious metal alloys for use in dentistry and since 2004 he has held the “Processing technologies” course at the Bicocca University of Milan in the ambit of the “Goldworking Sciences and Technologies” degree course. He is quality and R&D Manager at 8853 SpA in Pero, producer of precious metal semi-finished products and dental alloys. He has been a frequent speaker at the “Santa Fe Symposium” gold jewellery technology conference in Albuquerque and at the JTF in Vicenza.

Although the process of hot working metal dates back to the mists of time, it has only been sufficiently understood in the last 40 years. The greater workability of metals and alloys at high temperature is due to the quick change in microstructure of which dynamic re-crystallization is one of the main aspects. Probably due to the ease with which precious metals can be cold worked at room temperature, it is hard to find information on hot working. However, the difference in results between cold working followed by annealing and hot working is interesting and useful to know in order to improve product quality. This work aims at giving a general and simplified description of the micro-structural phenomena, like dynamic recovery and dynamic re-crystallisation, that occur during hot working. The advantages of hot working on some precious metals alloys will be illustrated by means of practical examples that will include an analysis of the microstructures.

DYNAMIC RE-CRYSTALLISATION AND THE HOT WORKING OF PRECIOUS METALS

INTRODUCTION

Although the process of hot working metal dates back to the mists of time, it has only been sufficiently understood in the last 40 years. Fundamental studies have been carried out on materials with widespread technological use, such as steels or aluminium alloys. However, very little information about the hot working of precious metal alloys is actually available. This is because hot working is not such a common process in jewellery making. As a matter of fact, the cold working of precious metals alloys is, in general, easy, the only complication being the need to anneal between the various stages of hardening. Furthermore, the ingots are generally small in size, which is a problem if the alloy needs to be kept hot during working. Hot working procedures require some special equipment, such as special rolls, suitable handling devices, and the chance of alloy contamination is higher. All this additional effort is hard to justify. However, some uses of hot working are known. The hot extrusion of precious metals is a well known process for wire, rod and tube production[1]. Hot stamping of gold alloys is sometime performed[2]. Hot forging is used during the mokume gane process[3].
In hot working, alloys are deformed at temperatures at which no hardening occurs and the alloy is resoftened continuously during working. Corti gave a basic description of hot working in his 2010 Santa Fe Symposium presentation[4]. The greater workability of metals and alloys at high temperature is due to the quick change in microstructure of which dynamic re-crystallisation is one of the main aspects. The difference in results between cold working followed by annealing and hot working is interesting and useful to know in order to improve product quality or set up new working procedures. This work aims at giving a general and simplified description of the micro-structural phenomena that occurs during hot working. The effect of hot working on the microstructures of some precious metals alloys will be shown by means of practical examples.

DYNAMIC RECRYSTALLISATION

The mechanisms by which alloys repair the structural damage caused by mechanical deformation have been described many times in the past[5,6,7,8] and are well detailed in literature[9,10]. These repair mechanisms are thermally activated, which means that the deformed alloy must be re-heated or, more precisely, annealed. During annealing the material undergoes two distinct processes: recovery and static re-crystallisation. The recovery paves the way for re-crystallisation. During recovery the deformed grains retain their identity, while the density and distribution of crystal defects change. Looking at the alloy microstructure through optical metallography, it is hard to understand if recovery has actually occurred. In the subsequent re-crystallisation, the crystal orientation of any region in the deformed grains changes, even more than once. A population of new grains is nucleated and grows at the expense of the deformed structure, until the latter is totally consumed. There are many “laws of re-crystallisation” but the four most important are:

1) a critical minimum deformation ε_r of the alloy is necessary to initiate re-crystallisation;
2) the smaller the degree of deformation, the higher the temperature required for re-crystallisation needs to be;
3) increasing the annealing time decreases the temperature required for re-crystallisation;
4) the grain size, after annealing, depends on the degree of deformation and, to a lesser measure, on the annealing temperature (being smaller the greater the degree of deformation and the lower the annealing temperature).

The process described by these laws is of central importance in the processing of metals and alloys for two main reasons. The first is due to the need to restore ductility in order to continue working, and the second derives from the need to control the grain structure of the final product on which many of its properties depend.

When alloys deform at elevated temperatures (more than 50% of the absolute melting temperature), hardening tends to be counteracted by recovery processes. If the recovery balances hardening entirely, stability is achieved and, if it can be maintained, considerable deformation can be performed before the material fractures. In some metals or alloys in which recovery is less rapid, certain conditions of stress and deformation temperature can result in the accumulation of sufficiently high local differences in dislocation density to nucleate re-crystallisation during deformation. This re-crystallisation is referred to as dynamic re-crystallisation[10,11,12,13,14].

The most important “laws of dynamic re-crystallisation” are:

1) dynamic re-crystallisation starts when a critical deformation $\square_c$ is achieved at a high temperature (above about 50% of the absolute melting temperature) but often the temperatures are relatively lower temperatures compared to those associated with static re-crystallisation. This critical deformation is usually higher than that needed to initiate static re-crystallisation ($\square_r$);
2) the re-crystallised grain size is uniquely determined by the applied stress and it is highly sensitive to the deformation rate and less affected by deformation temperature;
3) when deformation is halted, re-crystallisation does not stop immediately but continues at a gradually decreasing rate (meta-dynamic re-crystallisation).
4) dynamic re-crystallisation is repetitive by nature. This means that even when it is stable, there will be grains which have just re-crystallised, grains which have been deformed and are just about to re-crystallise again, and a spectrum of grains between these two limiting conditions all within different local regions.

Although dynamic re-crystallisation is a very complex subject since it depends on the many micro-structural characteristics of the alloy, it has been studied in detail in regard to some technological materials but very little information is currently available for precious metals. In order to have a very basic knowledge of what happens when a precious alloy is deformed at high temperature, some simple experiments have been performed.

MATERIALS AND METHODS

Deformation at a high temperature was applied by hammering the samples with a small forging hammer (figure 1) with a hammer weight of 40 Kg (88 lb).

Figure 1.

Forging hammer used to harden all the samples.

The hammer and anvil were flame-heated to about 250 °C (482°F) before starting the experiment.

Hot hammering was performed on two bars, the first made of Alloy 1 (95Pt-5Cu) (Table 1) and the second of Alloy 2 (sterling silver). The sterling silver bar was 24 x 24 mm in section and 1060 g in weight, while the platinum bar was 20 x 20 mm in section and 1300 g in weight.

The 95Pt-5Cu bar was heated using an oxygen-propane torch at approximately 900 °C (1652 °F) and then hammered at about 12 x 12 mm (64% reduction in section). During the procedure, the hammering was suspended once and the bar was re-heated with the torch to maintain the high temperature. When the final section was attained, the bar was annealed again by torch, cooled in water and then drawn at a section of 10 x 10 mm in 4 passages.

In order to compare hot and cold working procedures, a second 95Pt-5Cu bar, 20 x 20 mm in section, was cold rod milled down to 12 x 12 mm, annealed in the furnace at 900 °C (1652 °F) for 25 minutes and drawn to 10 x 10 mm in 4 passages.

A temperature of 900°C (1652 °F) was chosen for hot hammering so that it was equal to the furnace annealing performed on the second bar, the data for which were already available from past analyses. Annealing temperatures for platinum alloys are usually higher (1000 – 1200 °C / 1832 – 2192 °F).

The transversal sections of both platinum bars were compared by optical metallography.

The sterling silver bar was heated in a furnace to 700 °C (1292°F) for 30 minutes before hammering began. Hammering was only performed along half the length down to 11 mm (54 % reduction in thickness) in about 7 seconds. Then the bar was immediately cooled in water. The other end of the bar was then cold hammered in the same way down to a thickness of 11 mm. Figure 2 shows the bar at the end of the procedure. The transversal section of the bar was observed by optical metallography on both hammered sides and in the as cast section. The as cast section was also observed after thermal treatment at 700°C (1292°F) for 30 minutes.

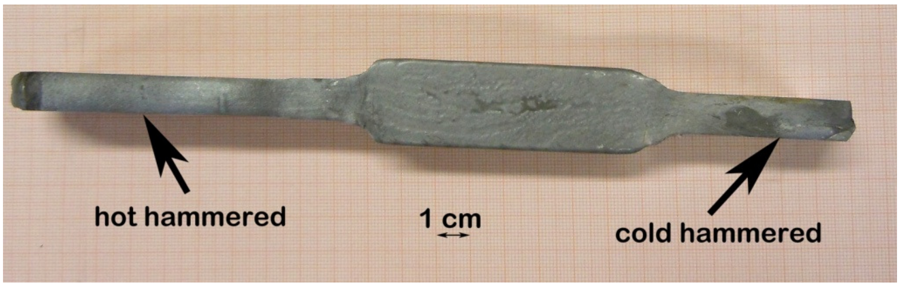

Figure 2.

Sterling Silver bar after hammering. The bar was annealed at 700°C for 30 minutes in the air and then immediately hot hammered on the left side. Then it was cooled in water and cold hammered on the right side.

In order to highlight some possible effects of hot hammering on small samples, alloys 2, 3 and 4 (Table 1) were investment cast in the shape of buttons: discs 15 mm in diameter and 3.5 mm in thickness (figure 3).

Three buttons were cast for each alloy. Of course the small size made it difficult to keep the sample hot during deformation. For this reason deformation was applied quickly, hammering the sample with just a few strokes in about two seconds with the forging hammer. Then the sample was immediately cooled in water.

One sample for each alloy was heated by oxygen-propane torch at the highest possible temperature according to the alloy melting range. An approximate temperature check was carried out using an optical pyrometer during flame heating. Hammering was carried out to a final thickness of 2 mm (43% reduction in thickness). The second sample of each alloy was cold hammered at 2 mm of thickness and was used to compare its microstructure to the hot hammered one. The third sample, if needed, was studied by optical metallography in order to characterise the as cast microstructure.

The Vickers micro-hardness was measured for each sample using a load of 200 gf. The hot hammered button in sterling silver was also examined by SEM/EDS.

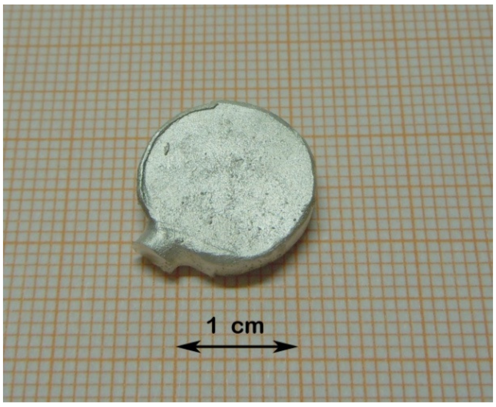

Figure 3. As cast appearance of the buttons made of different alloys.

Alloy	Composition (Weight %)
1	95 Pt – 5 Cu
2	92.5 Ag – 7.5 Cu
3	95 Pt – 5 Ru
4	75 Au – 8 Ni – 14 Cu – 3 Zn

Table 1. The table lists the different alloys studied in this work.

RESULTS AND DISCUSSION

Hot working precious metals is a rather uncommon process because it is generally unnecessary in the goldsmith field. Furthermore, the more common methods of performing hot working produce a poor surface finishing, the possible formation of an oxide scale and poor control of the final dimension. However, the following examples show that it has some interesting effects on microstructures, which may be useful.

1. Platinum alloy bars.

Figure 4 shows the microstructure of a Pt 95 Cu 5 bar, cold rod milled and cold drawn to the final size of 10 x 10 mm. The micrography shows the presence of internal voids and cracks. The microstructure is typical of deformed columnar grains. To better understand this aspect, figure 4 should be compared to figure 5 which represents the microstructure of the bar in the as cast conditions. In this figure, the columnar grains are well defined and not deformed by the rod milling process. Many gas pores are also visible which appear to be crushed during the rod milling in figure 4. The material has torn in the central area due to the presence of some of these pores and casting defects.

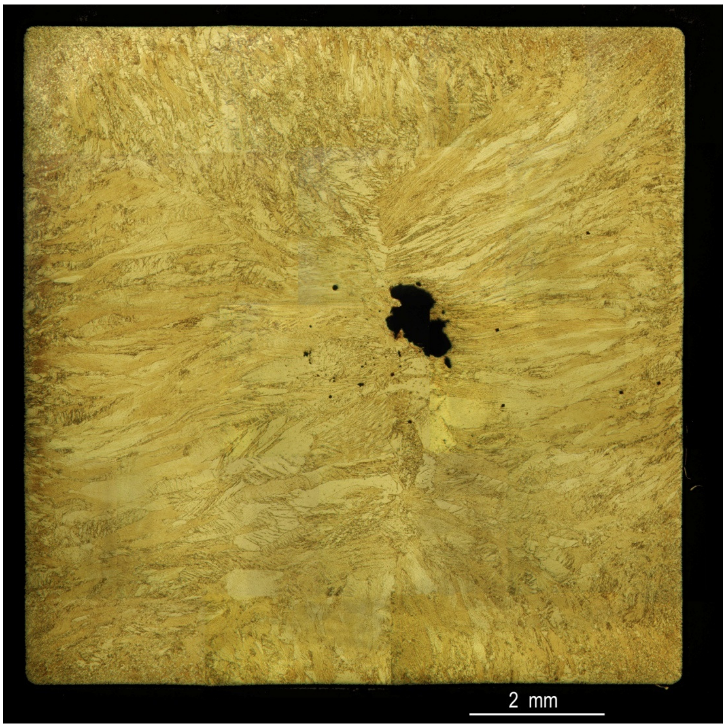

Figure 4. ***95 Pt- 5Cu. 208 HV_{200}.***

Transversal section of the bar, cold rod milled at 12 x 12 mm, annealed in the furnace at 900°C for 25 minutes and cold drawn at 10 x10 mm. The microstructure shows the as cast columnar grains deformed by the milling and drawing procedure. A tear is visible in the central area of the bar and some small pores are scattered along the section. To better understand the effects of the hardening on the microstructure, compare this image with figure 5 which refers to the as cast conditions.

It is not possible to see signs of re-crystallisation by looking at the microstructure in figure 4. However, with increased magnification, some partial re-crystallisation can be seen (figures 6 and 7) distributed irregularly along the section. The finished bar has voids and cracks inside, which are expected if we look at the wavy external surface (figure 8).

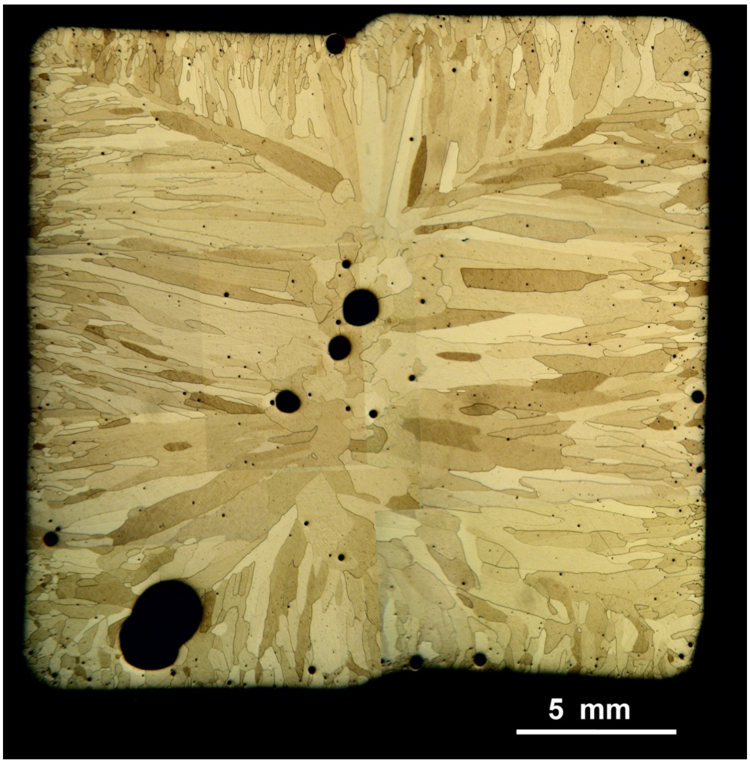

Figure 5.

95 Pt- 5Cu. 130 HV_{200}.

Transversal section of the bar in the as cast condition. This microstructure is typical of the original condition of both the cold and hot worked bars studied in this work. Columnar grains and gas pores are visible. Compare it with the microstructure seen in figure 4, after cold working, furnace annealing and final drawing. Notice the different magnification between the two figures.

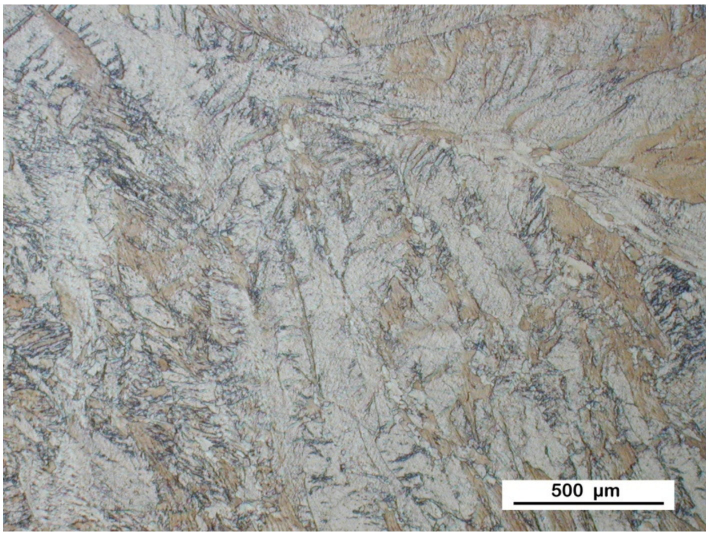

Figure 6. ***95 Pt- 5Cu. 208 HV_{200}.***

Detail of the microstructure seen in figure 4.

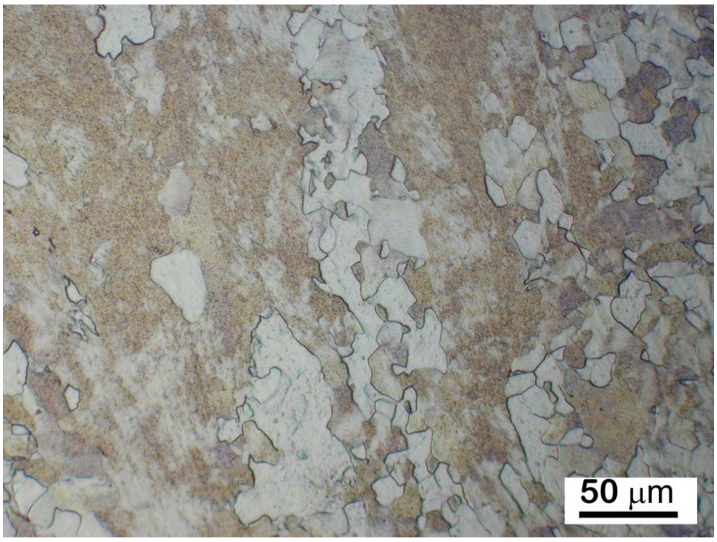

Figure 7. ***95 Pt- 5Cu. 208 HV₂₀₀.***

Detail of the microstructure seen in figure 4. The effect of the static re-crystallisation due to annealing can be seen. Annealing was carried out before the final drawing. Re-crystallisation is not uniformly distributed along the section of the bar.

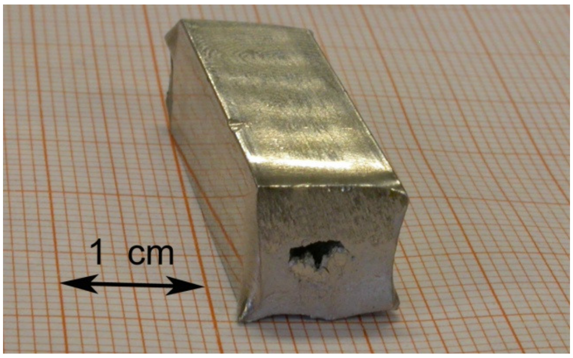

Figure 8.

95 Pt- 5Cu. 208 HV₂₀₀.

Piece of the bar seen in figure 4 which shows a wavy external surface after drawing due to the internal cracks and voids.

Figure 10 shows the microstructure of the Pt 95 Cu 5 bar hot hammered and cold drawn down to 10 x 10 mm. The microstructure appears more homogeneous than the in previous case. Internal pores and cracks are absent. The as cast columnar grains have been substituted by smaller grains (figure 11). The microstructure in figure 10 must be compared with that in figure 4, which is taken at the same magnification.

As regards the hardness, data are reported in table 2 and in each figure caption. It is interesting to note that there are practically no differences between the hot hammered and the cold worked and annealed platinum bar despite the very different microstructures and the internal ruptures that are typical of the cold working procedure.

It is very important to note that the above-described hot hammering procedure involves many different metallurgical processes. Not only can the alloy recover and re-crystallise during the deformation produced by the hammer, it can also go through static recovery and re-crystallisation during re-heating by torch (figure 9). Furthermore, metallurgists consider a third kind of re-crystallisation: the meta-dynamic re-crystallisation which immediately follows the cessation of hot deformation[10]. In this example meta-dynamic re-crystallisation takes place between one hammer stroke and the next.

In general, it can be said that the hot working method gives a better final microstructure and, above all, stops ruptures because it produces the sintering of voids present in the as cast condition (diffusion bonding). It is well known that dynamic re-crystallisation plays a leading role in effectively sintering pores[10]. It is worthwhile underlining that the benefit of platinum alloy hot working is well known. The example below shows that dynamic re-crystallisation must be the reason for these benefits by giving support in transforming into a microstructure with improved characteristics.

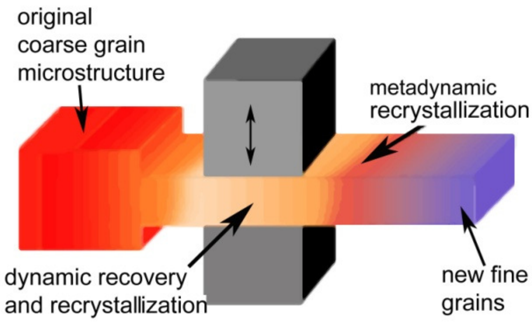

Figure 9.

Hot hammering is a complex process involving different kinds of recovery and re-crystallisation: dynamic at the moment of the hammer stroke, meta-dynamic immediately after the hammer stroke and static when the bar is re-heated by torch.

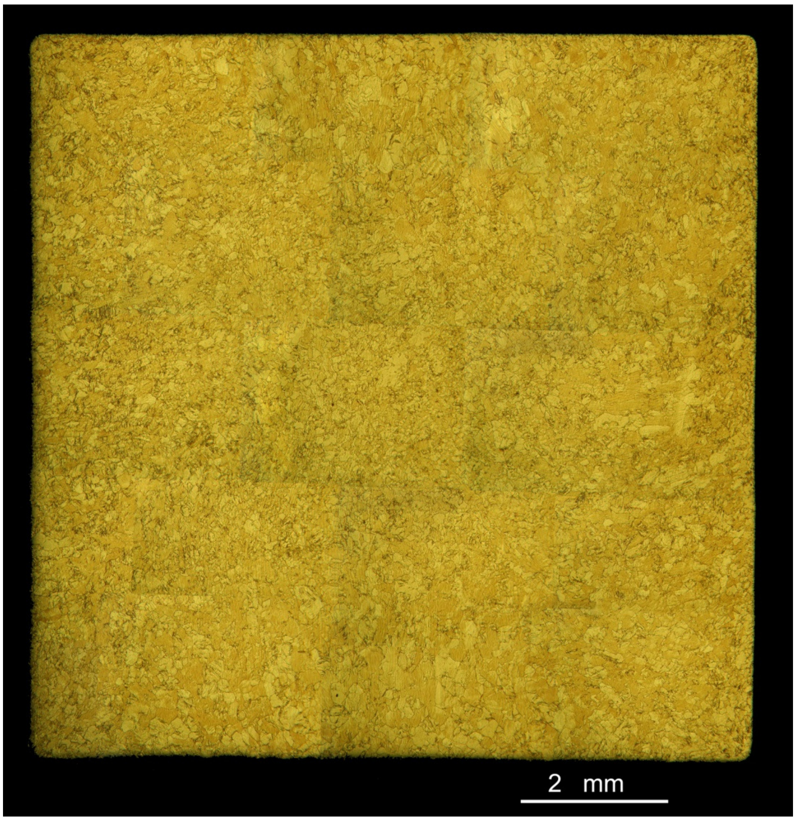

Figure 10.

95 Pt- 5Cu. 200 HV$_{200}$.

Transversal section of the bar, hot hammered at 12 x 12 mm, annealed by torch and cold drawn at 10 x10 mm. The microstructure is more homogeneous than in the case of cold rod milling seen in figure 4, with smaller and equiaxed grains. There are no voids.

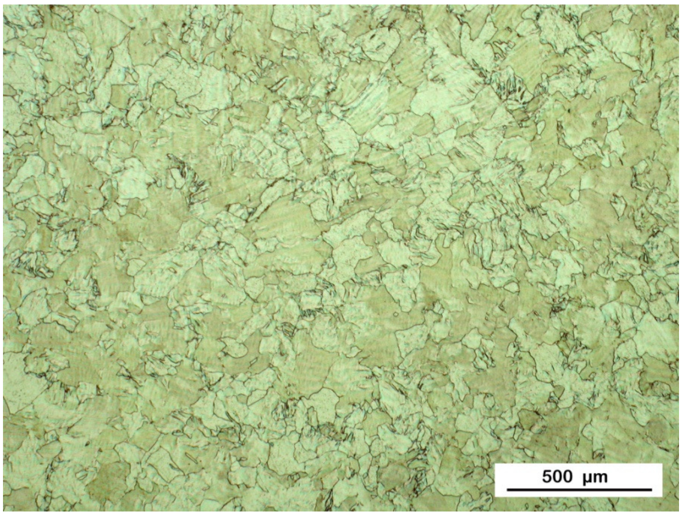

Figure 11.

95 Pt- 5Cu. 200 HV200.

Detail of the microstructure seen in figure 10. The hot hammering has produced a grain refinement due to the support of dynamic re-crystallisation. Compare to figure 6.

2. Sterling Silver bar.

Figure 12 shows the microstructure of a sterling silver bar in the as cast condition. The alloy shows a strong microsegregation[15]. The microstructure, after annealing at 700°C for 30 minutes and air cooling, is seen in figure 13. This condition represents the situation before cold hammering. Here, the microsegregation and the hardness are reduced in comparison to the as cast alloy. Optical metallography shows a subtle difference between the hot hammered (figures 14 and 15) and the cold hammered microstructures (figures 16 and 17). However, the onset of re-crystallisation is visible in figure 16, and the hardness (table 2), practically equal to the as cast value, confirms that re-crystallisation has commenced. Re-crystallisation has occurred during hot hammering and the very short time preceding the plunging of the bar in water. What can be seen here also explains the shorter time (and less energy) necessary to hot hammer the bar in comparison to the cold hammering performed on the other side of the silver bar. Figure 18 shows what happens if the hot hammered alloy is subject to static annealing at 650°C for 10 minutes: new and larger re-crystallised grains have grown.

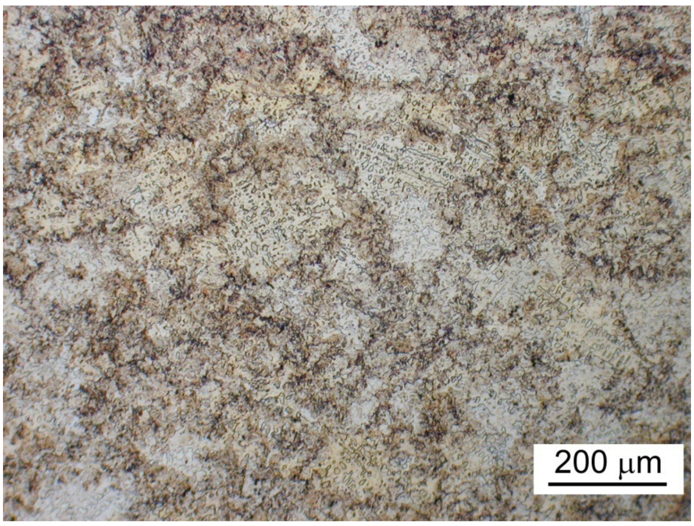

Figure 12. ***92.5 Ag- 7.5Cu. 81 HV200.***

Microstructure of the as cast sterling silver bar. The alloy shows a strongly segregated microstructure.

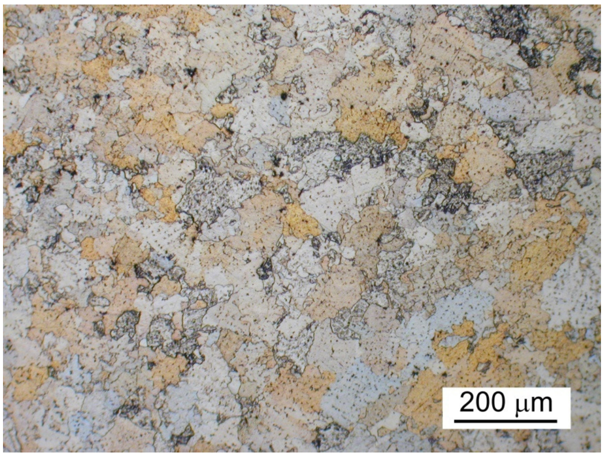

Figure 13. **92.5 Ag- 7.5Cu. 55 HV_{200}**

Microstructure of the sterling silver bar after annealing at 700°C for 30 minutes. The segregation is reduced together with the hardness. This microstructure represents the conditions preceding the cold hammering.

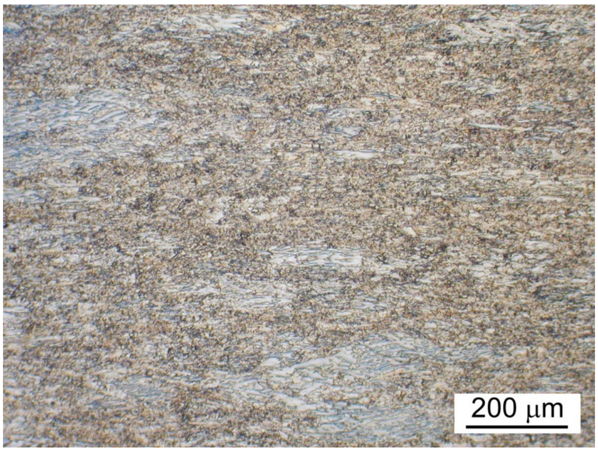

Figure 14. **92.5 Ag- 7.5Cu. 85 HV_{200}**

Microstructure of the sterling hot hammered silver bar. Transversal section of the bar. The alloy shows a fibrous microstructure, apparently not re-crystallised. However, the hardness is practically equal to the value measured in the as cast condition.

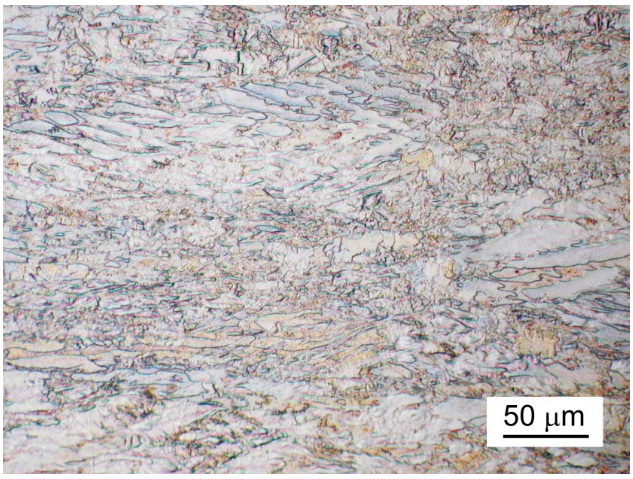

Figure 15. **92.5 Ag- 7.5Cu. 85 HV_{200}**

Detail of the microstructure seen in figure 14. At this magnification, the onset of re-crystallisation can be seen. It is recognisable from the very thin fragmentation of the microstructure: compare to figure 17.

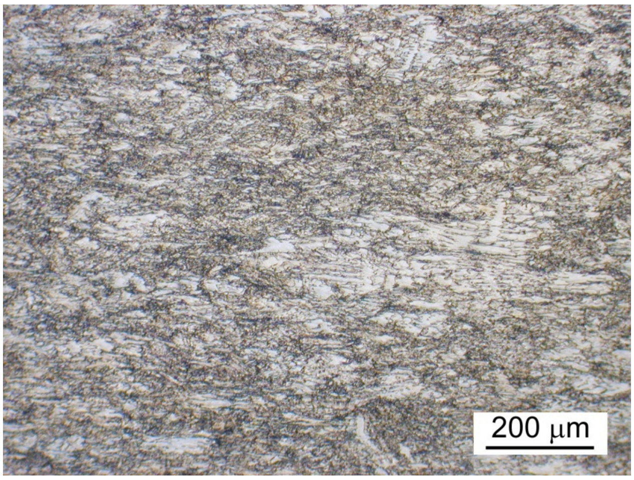

Figure 16. ***92.5 Ag- 7.5Cu. 137 HV₂₀₀***

Microstructure of the sterling cold hammered silver bar. Transversal section of the bar. The alloy shows a fibrous microstructure.

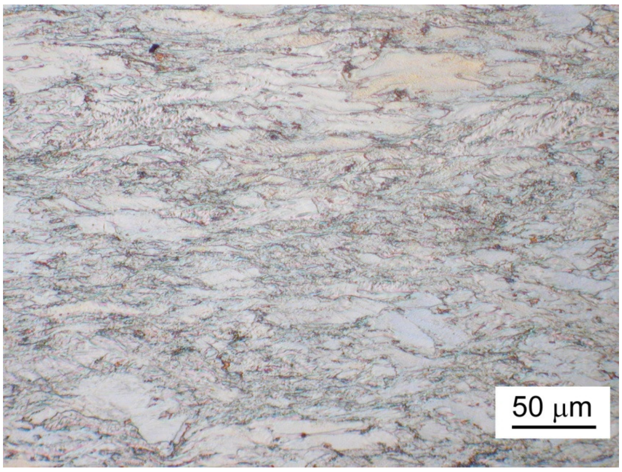

Figure 17. ***92.5 Ag- 7.5Cu. 137 HV₂₀₀***

Detail of the microstructure seen in figure 16. Notice the subtle differences with the microstructure of the hot hammered alloy (figure 15)

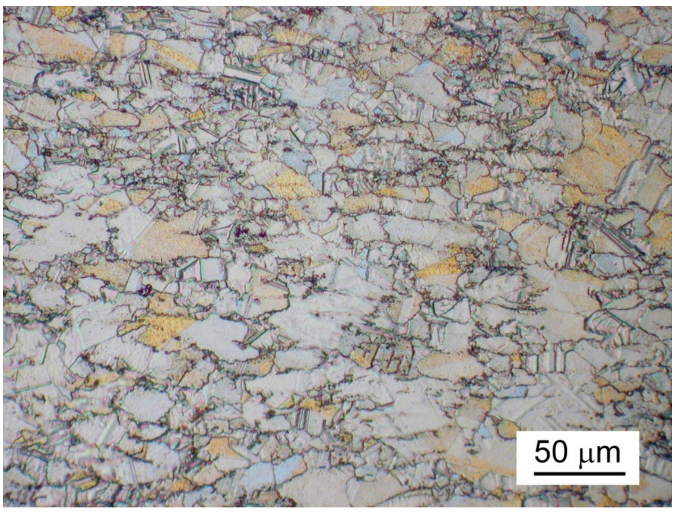

Figure 18. ***92.5 Ag- 7.5Cu. 63 HV₂₀₀***

The figure shows the evolution of the microstructure seen in figure 15 after static annealing at 650°C for 20 minutes. New grains are visible due to the static re-crystallisation.

3. Buttons of different alloys.

Figure 19 shows the as cast segregated microstructure of alloy 2, typical of sterling silver. Notice the great difference in microstructure with that seen in figure 12. The difference is due to the different solidification and cooling rate. After hot hammering, new small grains are visible around the dark islands which are the eutectic structural constituent of the alloy (figure 20 and 21). These new grains have a greater hardness if compared with the surrounding alloy matrix. Micro-hardness measurements give a mean value of 150 HV_{15} for the new grains and 105 HV_{15} for the matrix. Furthermore, the EDS micro-analysis shows that the new grains are richer in copper than the matrix. In spite of all these observations, it is difficult to give a univocal description of this evolution in the microstructure. It may be possible that the new grains are the results of dynamic re-crystallisation which typically nucleate on inclusions or second phases[10,14]. The greater copper content of these new grains is evidence of atomic diffusion from the eutectic islands. This diffusion is favored during dynamic re-crystallisation. Comparison with the cold hammered sample (figure 22) shows the absence of new re-crystallised grains in this case. This absence justifies the slightly lower hardness of the cold hammered alloy.

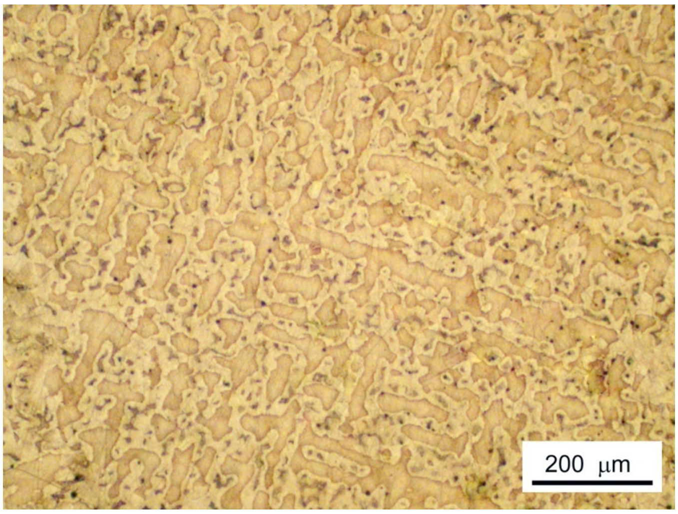

Figure 19. ***92.5 Ag- 7.5Cu. 62 HV_{200}***

Microstructure of the as cast sterling silver button. The alloy shows a segregated microstructure.

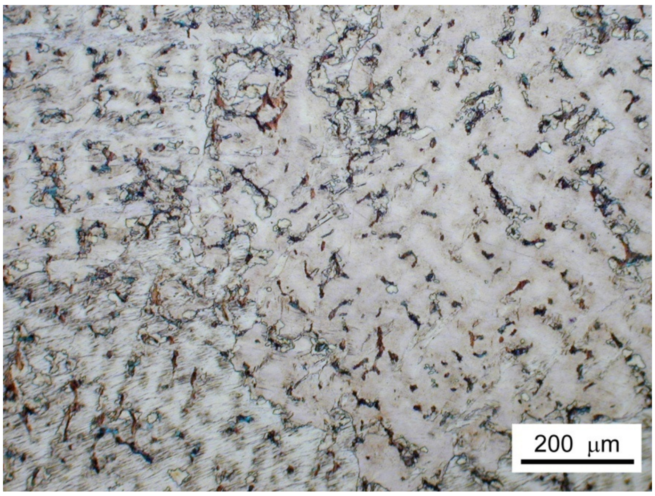

Figure 20. ***92.5 Ag- 7.5Cu. 116 HV_{200}***

Microstructure of the hot hammered sterling silver button. The alloy shows a deformed microstructure with small new grains around darker small islands (the eutectic structural constituent). Figure 21 shows more detail.

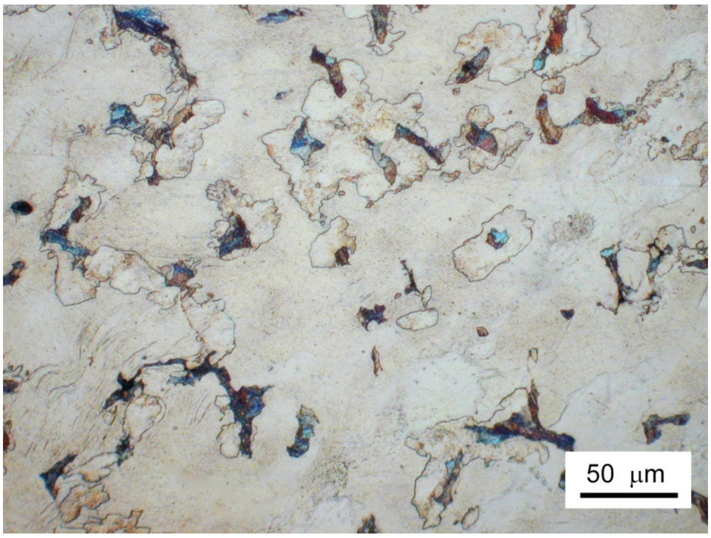

Figure 21. ***92.5 Ag- 7.5Cu. 116 HV$_{200}$***

Detail of figure 20. The alloy shows a deformed microstructure with small new grains around the darker small islands (the eutectic structural constituent).

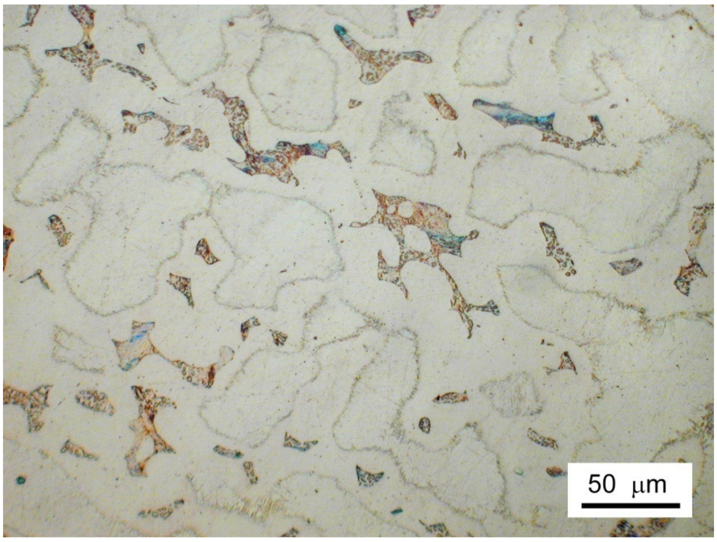

Figure 22. ***92.5 Ag- 7.5Cu. 109 HV$_{200}$.***

Microstructure of the cold hammered alloy. Notice the absence of new re-crystallised grains. Compare to figure 21.

Figure 23 shows the microstructure of the hot hammered Pt95-Ru5 alloy, while figure 24 refers to the cold hammered sample. The grains of the hot hammered sample appear more fragmented in comparison to the grains of the cold hammered alloy. Even though the difference between the two microstructures is evident, it is difficult to give an exhaustive description of the reason. We know that the electrochemical etching used to highlight the microstructure is particularly effective when there is a high density of reticular defects, such us grain boundaries. Therefore, a possible interpretation of the microstructure seen in figure 23 is that here, the deformation is characterised by grain subdivision into differently oriented regions. Hot working, performed on this sample in just a few seconds, supported this change of microstructure, preparing the material for re-crystallisation. Notice that the hot hammered sample has a slightly lower hardness compared to the cold hammered one.

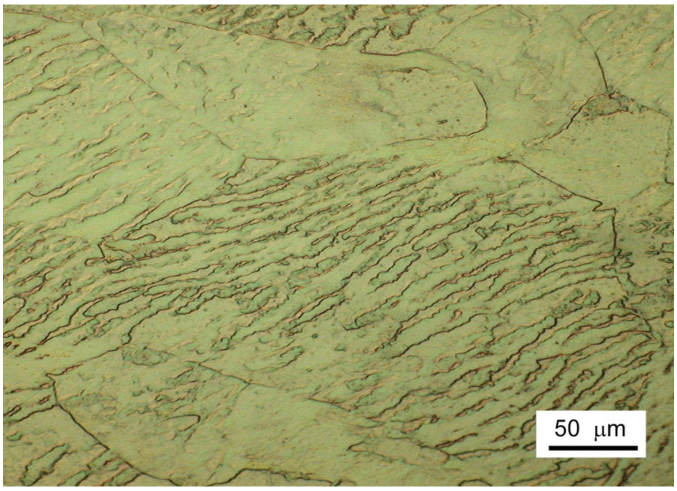

Figure 23. ***95Pt-5Ru. 174 HV200.***

Hot hammered alloy. Transversal section of the button. The grain is subdivided into differently oriented regions approximately arranged in parallel bands.

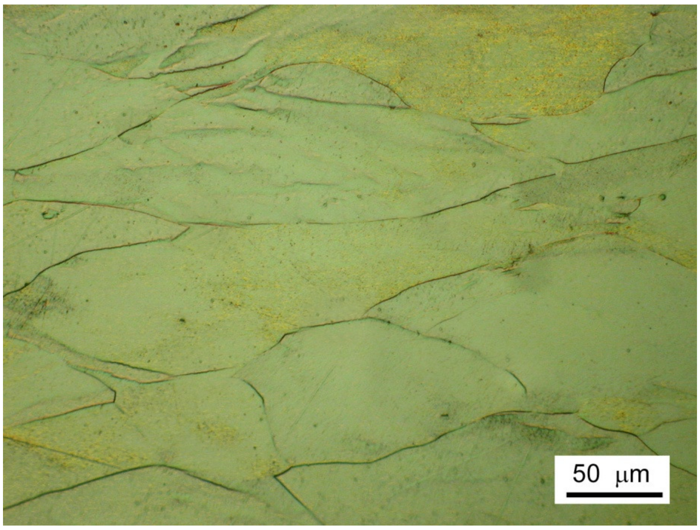

Figure 24. ***95Pt-5Ru. 194 HV200.***

Cold hammered alloy. The grain shows a deformation due to hardening in comparison to the equi-axed shape typical of the as cast sample.

Figures 25 and 26 show the microstructure of hot hammered, 18-carat, white gold, while figure 27 shows the microstructure of the cold hammered sample. The comparison of figures 25 and 27 highlight the reduction in the microsegregation and the presence of small grains, less than 20 microns wide, in the hot worked material. Figures 25 and 26 show that small grains, with a size ranging from just a few microns to 30 microns, are growing along the grain boundaries. This behaviour is known as the necklace mechanism of dynamic re-crystallisation[12,16]. The occurrence of dynamic re-crystallisation leads to grain refinement at high strain rates and under hot working conditions. Such refinement is initiated at the grain boundaries of the existing microstructure and proceeds with the nucleation of successive "necklaces" of new grains through a process referred to as "necklacing". Figure 28 shows a simplified model of this process.

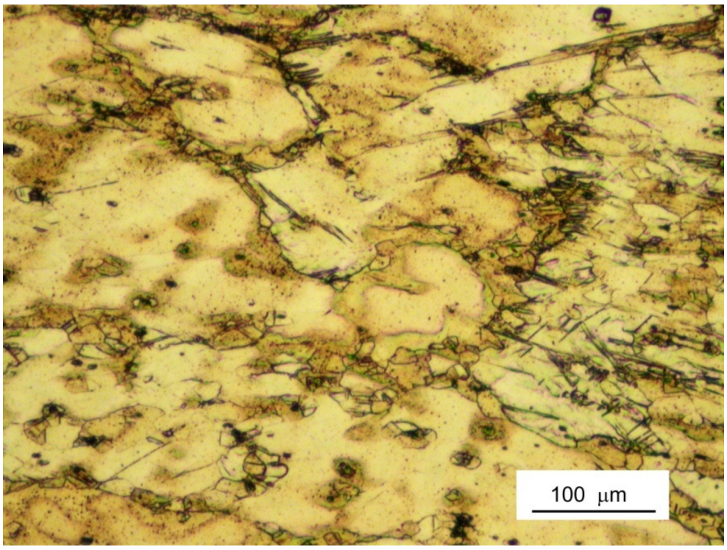

Figure 25. ***75 Au – 8 Ni – 14 Cu – 3 Zn. 236 HV200.***

Hot hammered alloy. The microsegregation typical of the as cast sample (figure 27) is reduced and small new grains are visible along the grain boundaries.

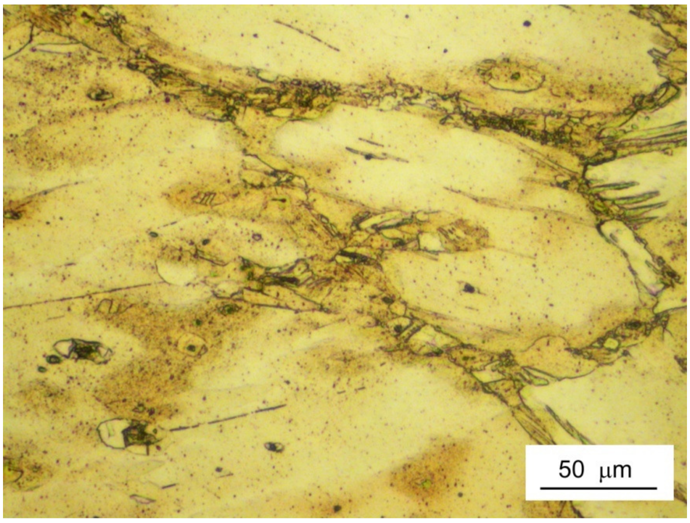

Figure 26. ***75 Au – 8 Ni – 14 Cu – 3 Zn. 236 HV200.***

Detail of figure 25. Small grains with sizes ranging from just a few microns to 30 microns are visible along the grain boundaries due to a process referred to as "necklacing" that is typical of dynamic re-crystallisation.

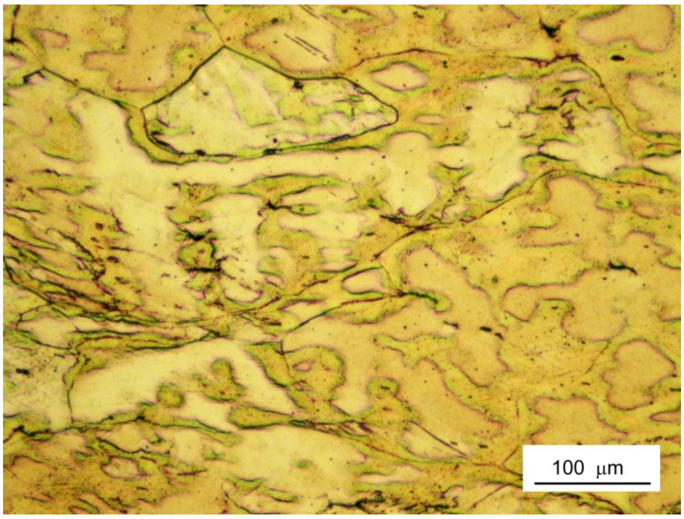

Figure 27. ***75 Au – 8 Ni – 14 Cu – 3 Zn. 281 HV200.***

Transversal section of the cold hammered sample. The strong microsegregation characterising the as cast microstructure is still well visible. It looks like islands with rounded borders distributed inside the grains

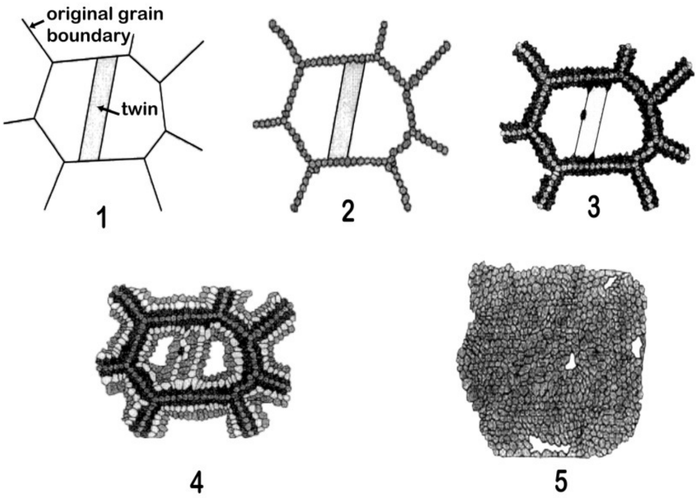

Figure 28.

Dynamic re-crystallisation evolution with the necklace mechanism. New grains start to grow along the original grain boundaries forming a necklace of grains. New shells of grains grow while the hot deformation continues.

Alloy		Hot hammered	Cold hammered	rod milled	As cast
1	95Pt – 5 Cu bar 20x20 mm	200 ± 9		208 ± 13	130 ± 4
2	Sterling silver button	116 ± 7	109 ± 6		62 ± 4
2	Sterling silver bar 24x24mm	85 ± 3	137 ± 8		81 ± 2
3	95 Pt – 5 Ru button	173 ± 6	194 ± 13		125 ± 5
4	18-carat, white gold button	236 ± 10	281 ± 19		200 ± 4

Table 2.

The Vickers micro-hardness measured with a load of 200 gf is given for all the analysed samples.

CONCLUSION

The fact that it is difficult to find information about hot working precious metals in literature is due to the uncommon use of this process. However, the simple experiments described herein have highlighted significant differences between cold and hot working of some precious metals alloys.

Pore sintering and grain refining of hot hammered 95Pt-5Cu was illustrated. Dynamic re-crystallisation of silver sterling during hot hammering was observed and required less time and energy than cold hammering. Small samples, in spite of the very short time during which they remain hot, show signs of dynamic re-crystallisation: necklace re-crystallisation in white gold, local re-crystallisation in sterling silver and grain subdivision in 95Pt5Ru alloy.

A fully comprehensive analysis of all the observed processes is not included in the aim of this work, but further investigation may be useful to improve the material workability and the quality of final products.

Dynamic re-crystallisation of precious metals is possible, but its widespread use.

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