МАТЕРИАЛОВЕДЕНИЕ И ТЕРМИЧЕСКАЯ ОБРАБОТКА МЕТАЛЛОВ

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OPTIMIZATION OF SOFTENING HEAT TREATMENT FOR THE Pt950Ru JEWELRY ALLOY

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Abstract. Platinum is one of the most precious metals in the world: 15 times rarer than gold and it is widely used in jewellery for its characteristic of unicity, incorruptibility, and colour neutrality.
The standard grade in the jewellery industry is Platinum 950‰. In fact, pure platinum jewels are easily scratched, due to the poor hardness linked with the high purity. Among all the commercial alloys available, the Pt-Ru system is the one characterized by the highest mechanical properties. Aim of this work is to find an ideal softening heat treatment, in term of operating time and temperature. The heat treatment must be able to satisfy two main needs: it must permit to complete recrystallization of the work piece, preparing the material for further severe deformation by restoring high ductility and an equiaxed grain distribution; and at the same time, the final average grain size must be fine, in order to avoid counter side aesthetic effects that occur when grain size go over the micrometric scale. Due to its rarity, in literature there are few information about the alloy under discussion. To find the optimal parameters, several samples had been heat treated at different times and temperatures. Then they had been analysed by optical microscopy and with micro hardness tests, providing microstructure images and hardness data. By using a combination of three different temperatures and three different times, significant differences were found between the various samples both in terms of mechanical properties and of grain size. The comparison of the collected data permit to better understand the behaviour of the alloy under recrystallization conditions. In the end, it was observed that the dynamics of recrystallization changes significantly with relative small changes of temperature and was found that the optimal treatment to obtain a fine recrystallized microstructure with the desired mechanical properties is at 1000°C for 15 minutes.

Keywords: Platinum alloy, softening heat treatment, jewellery, microhardness, microstructure.

Introduction

Platinum alloys represent the second most important production filed in the jewelry world, after gold alloys [1].

Despite gold, the most widespread standard for platinum jewels is platinum 950 (95 wt.%). This means that the addition of other metals can be done up to 5 wt.%, limiting the possibility of reinforcing the metal by alloying [2]. Object of the study is the platinum-5 wt.% ruthenium alloy, a widely used alloy for jewelry manufacture which has good values of hardness compared with other platinum alloys (as can be observed in the graph in **Fig.1**, without compromise ductility [3–4].

Besides investment casting, fine jewelry products can be manufactured also via mechanical processes: a forged bar is cold rolled and annealed, and subsequently machined, until the net shape product is obtained. This procedure allows a reduction of porosity [5]. In order to obtain a high deformation, after every step of cold rolling is necessary an annealing treatment, to recrystallize the structure and permit further deformation [6]. Unfortunately, there are few information in literature about platinum processing, and in particular about annealing parameters [7]. In detail only few works regarding the Pt-5% Cu alloys can be found [4,8]. The aim of this work is to study the role of time and temperature of the annealing process on the microstructure and the hardness of the alloy.

Experimental

The samples studied in the present work come from the industrial process for the production of platinum watchbands. An example of the final product can be observed in **Fig. 2**.

All the samples analyzed are derived from a single sample of platinum-ruthenium alloy in the annealed state, with homogeneous grain size and equiaxed distribution **(Fig. 3)**, and a hardness of 185 HV. The nominal composition of the alloy is reported in **Tab.1.**

Tab. 1

Composition of the Pt950Ru alloy

COMPOSITION	
Pt	95%
Ru	5%

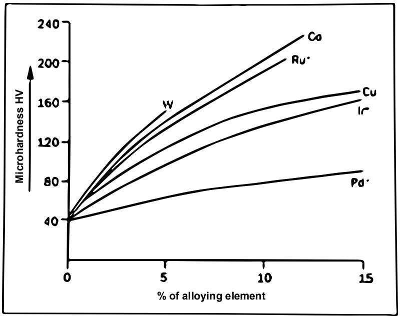

Fig.1. Micro-hardness evolution with different alloying elements in platinum alloys

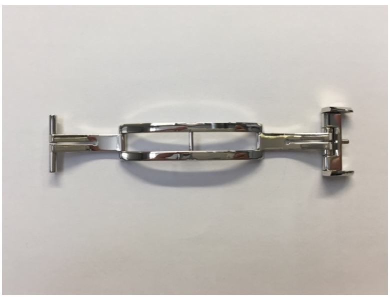

Fig. 2. Example of watchbands in Pt950Ru alloy at the end of the production cycle

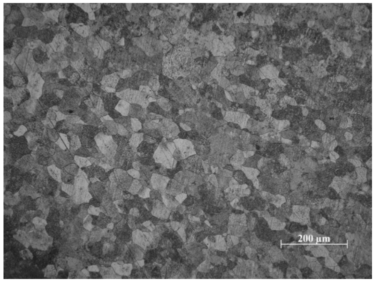

Fig. 3. Microstructure of the starting sample, 100x

The original thickness of 1,6 mm has been reduced to 0,48 mm by a cold rolling process, in order to achieve a reduction of 70%. This percentage of reduction was used for two main reason: it is in line with the common production processes, and allows recrystallization phenomena.

Once cold rolled, the material has been cut in several pieces. The pieces have been heat treated in a furnace with scheduled time and temperature. The low dimensions of the samples make almost negligible heating time of the single piece.

After the heating process, the samples have been quenched in ambient temperature water.

Later, the samples were polished with standard metallographic technique, and microhardness test have been conducted using microVickers 0.5 scale with a Leitz micro indenter.

The etching of grain boundaries has been performed by immersing the samples in boiling aqua regia for 15 minutes. The optical microscope (OM) observation was performed with a Leica DMRE microscope.

Results and discussion

The samples were heat treated at 950 °C, 1000°C and 1050°C for 10, 15 and 20 minutes and the results in term of microstructure and hardness properly evaluated.

In **Fig. 4** can be observed the optical micrographs of the samples heat treated at 950°C for different treatment times. It can be observed that at these temperatures no evident recrystallization phenomena occurs for 10 **(Fig. 4a)** and 15 **(Fig. 4b)** minutes. Only after 20 minutes **(Fig.4c)** recrystallization is partially observed. In all the treatment times the grains resulted still oriented in the rolling direction. Hardness values of the samples decrease to 200 HV **(Tab. 2)** due to the recovery process, but this is not sufficient to permit the subsequent sequences of plastic deformation.

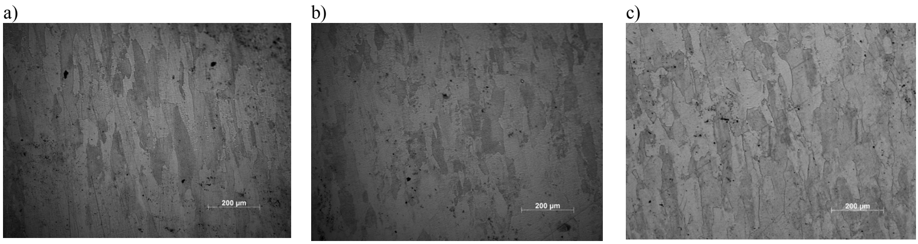

Fig. 4. 950°C and: a) 10 min; b) 15 min; c) 20 min. 100x

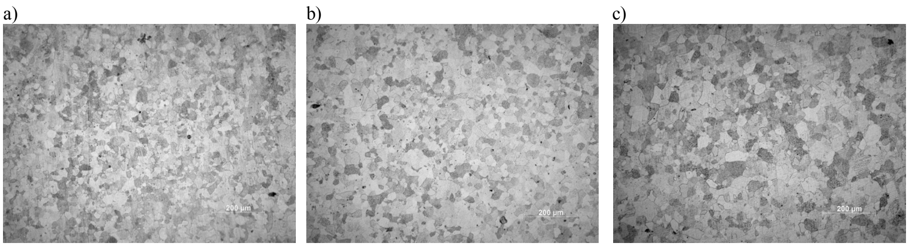

Fig.5. 1000°C and: a) 10 min; b) 15 min; c) 20 min. 100x

At 1050°C the kinetic of recrystallization result substantially faster than the 1000°C one, leading to an irregular grain growth as shown in **Fig.6**. For this reason, it was decided not to take into account a detailed analysis of the samples annealed for longer periods.

Tab. 2

Hardness values

HARDNESS VALUES (HV 0.5)			
	10 min	**15 min**	**20 min**
950°C	210	206	204
1000°C	150	140	135

Tab. 3

Grain size average values

GRAIN SIZE (µm)			
	10 min	**15 min**	**20 min**
950°C	Not recrystallized	Not recrystallized	Not recrystallized
1000°C	35	45	70

When heat treated at 1000°C, the sample completely recrystallizes in the first 10 minutes **(Fig.5a)**. The new grain is fine and equiaxed (average grain size 35 µm as reported in **Tab.3**), and it is not possible to recognize the orientation of the rolling process. After 15 minutes **(Fig.5b and Tab.3)** the average grain size results increased to 45 µm, and after 20 minutes **(Fig.5c and Tab.3)** it reaches an average value of 70 µm. Likewise, the grain growth, hardness decreased from 150 HV to 135 HV **(Tab.2)**, proving that the complete recrystallization occurred.

Fig.6. 1050°C and 10 min. 100x

The evolution of the mechanical properties, in terms of micro-hardness, with the treatment time is summarized in **Fig.7**. It can be observed both for 950 and 1000°C samples a decrease in the hardness in comparison with the reference value of the untreated sample. However, for the sample treated at 1000°C the decrease in the hardness in more significant. This behavior can be attributed to the fact that at 950°C only recovery occurs as confirmed by the micrographs previously reported. Instead at

1000°C complete recrystallization phenomena can be observed with the optimal mix in term of hardness and grain size obtained for a treatment of 15 minutes.

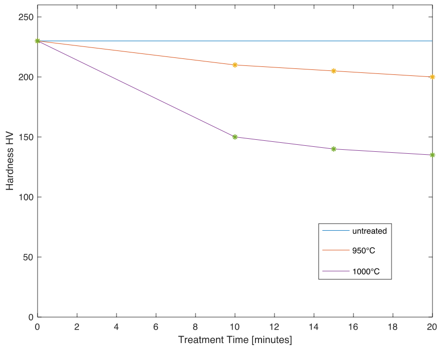

Fig.7. Evolution of the micro-hardness with the treatment time for the samples treated at 950 and 1000°C

Conclusions

From the comparison of the data collected it has been possible to identify the optimal conditions in terms of temperature and treatment time for an annealing softening treatment on a Pt950Ru alloy. In order to obtain the complete recrystallization during the annealing process, it is necessary to use a temperature of 1000°C: at this temperature the new grain originated is fine, equiaxed, and its growth is controllable. As regards the treating time, the 15 minutes sample showed an hardness slightly greater than the 20 minutes one (140 HV the former, 135 the latter), but a much finer microstructure. An image at higher magnification of the sample obtained with optimized treatment conditions can be observed in **Fig.8**.

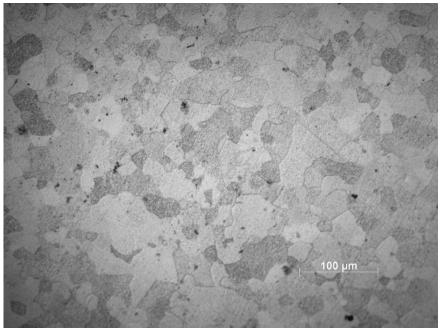

Fig.8. 1000°C and 15 min. 200x

References

1. Gold Demand Trends Q3 2016. World Gold Council, 8th November 2016
2. T. Biggs. The Hardening of Platinum Alloys for Potential Jewellery Application. Platinum Metals Rev., 2005, 49, (1), 2–15
3. G. Rakhtsaum. Platinum Alloys: A Selective Review of the Available Literature. Platinum Metals Rev., 2013, 57, (3), 202–213
4. K.M. Jackson, C. Lang. Mechanical Properties Data for Pt-5 wt.% Cu and Pt-5 wt.% Ru Alloys. Platinum Metals Rev., 2006, 50, (1)
5. S. Tedesco. Manuale di tecnologia e lavorazione del platino, Atti del congresso Tecno Platino, Vicenza 8 Luglio 1999.
6. J.C. Wright. Jewellery related properties of platinum. Platinum Metals Rev., 2002, 46, (2)
7. G. Normandeau, D. Ueno. Understanding Heat treatable Platinum Alloys. "The Santa Fe Symposium on Jewelry Manufacturing Technology 1999", Proceedings of the 13th Santa Fe Symposium in Albuquerque, New Mexico, USA, ed. D. Schneller, Met-Chem Research, Albuquerque, New Mexico, USA, 1999, 73–103
8. C. Mshumi, C. Lang. Strenghtening of Platinum-5wt% Copper by Heat Treatment.. Platinum Metals Rev., 2007, 51, 78–82

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ОПТИМИЗАЦИЯ РЕЖИМА СМЯГЧАЮЩЕЙ ТЕРМООБРАБОТКИ ДЛЯ ЮВЕЛИРНОГО СПЛАВА Pt950Ru

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Аннотация. Платина является одним из наиболее ценных металлов в мире. Платина встречается в 15 раз реже золота и находит широкое применение в ювелирном деле благодаря своей уникальности, прочности и нейтральному цвету. Стандартный сорт платины, применяемый в ювелирной отрасли, - это платина 950 пробы. На самом деле изделия из чистой платины легко повредить из-за их недостаточной твердости, связанной с высокой степенью чистоты металла. Среди всех имеющихся промышленных сплавов наилучшими механическими свойствами обладает система Pt-Ru. Целью настоящей работы является определение идеального режима смягчающей термической обработки с точки зрения времени и температуры. Такая термообработка должна выполнять две основные задачи: способствовать завершению процесса рекристаллизации образца и подготовке материала к последующему этапу интенсивной деформации за счет восстановления свойств повышенной вязкости и равноосной микроструктуры; и в то же самое время способствовать образованию мелкозернистой структуры для исключения обратного эстетического эффекта, возникающего, когда размер зерен перестает измеряться в микрометрах. Из-за его редкости в литературе находятся лишь немногие упоминания о рассматриваемом сплаве. Для определения оптимальных параметров термообработки несколько образцов были подвергнуты термической обработке при различной температуре и различном времени обработки. После этого образцы были изучены на оптическом микроскопе и микротвердомере, с помощью которых были получены изображения микроструктуры и данные о твердости. Образцы были подвергнуты трем различным температурно-временным режимам термообработки. Были обнаружены значительные различия между образцами по механическим свойствам и размеру зерен. Сравнение полученных данных помогает лучше понять поведение сплава в условиях рекристаллизации. В итоге было установлено значительное изменение динамики процесса рекристаллизации при относительно небольших изменениях температуры. Установлено, что оптимальный режим термообработки, позволяющий получить мелкую рекристаллизованную микроструктуру и требуемые механические свойства, подразумевает обработку при температуре 1000°С в течение 15 мин.

Ключевые слова: платиновый сплав, смягчающая термообработка, ювелирные украшения, микротвердость, микроструктура

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