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MECHANICAL PROPERTIES AND MICROSTRUCTURE CHARACTERISATION OF Au-Pt DENTAL ALLOY

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Development of a dental alloy with high Au content is based on the ternary system of Au-Pt-Zn with a nominal composition of 86,9Au-9,9Pt-1,5Zn, and about 1,5 wt.% micro-alloying elements (In, Ir, Rh). The results analyses of different heat-treated states showed that the optimal mechanical properties and hardness of an Au-Pt-Zn alloy can be reached with combinations of heat treatment for 20 minutes at 723 K and then slowly cooling, if the alloy was annealed at 1223 K for 30 minutes and the water quenched. Research results confirmed that the microstructure of the Au-Pt-Zn alloy consists of two phases: α_1-phase rich in Au (main phase) and α_2-phase rich in Pt (minor phase). During XRD analysis and use of the Rietveld method, it was found that the α_1-phase content is about 98,5 wt.% while the content of α_2-phase is 1,5 wt.%. STA analyses show that the Au-Pt-Zn alloy has a solidus temperature of about 1292 K and a liquidus temperature of about 1412 K.

Key words: Au dental alloy, characterisation, microstructure, properties

Mehanička svojstva i mikrostrukturna karakterizacija Au-Pt dentalne slitine. Razvoj dentalne slitine s visokim sadržajem zlata zasnovan je na ternarnom sustavu Au-Pt-Zn s nominalnim sastavom 86,9Au-9,9Pt-1,5Zn i oko 1,5 mas. % mikrolegirajučih elemenata (In, Ir, Rh). Rezultati analize su pokazali da se u slučaju slitine odžarene kod 1223 K u trajanju od 30 minuta i zakaljena u vodi optimalna mehanička svojstva i tvrdoća Au-Pt-Zn slitine mogu postići kombinacijom toplinske obrade na 720 °C u trajanju 20 minuta s naknadnim sporim hlađenjem. Rezultati istraživanja su potvrdili da se mikrostruktura Au-Pt-Zn slitine sastoji od dvije faze: α_1-faze bogate sa zlatom (primarna faza) i α_2 - faze bogate s platinom (sekundarna faza). Tijekom XRD analize i primjenom Rietveldove metode utvrđeno je da je sadržaj α_1-faze oko 98,5 mas. %, dok je sadržaj α_2-faze iznosio oko 1,5 mas. %. STA analiza je pokazala da je Au-Pt-Zn slitina imala solidus temperaturu oko 1292 K, a likvidus temperaturu oko 1412 K.

Ključne riječi: Au dentalna slitina, karakterizacija, mikrostruktura, svojstva

INTRODUCTION

Gold alloys have been used in dentistry, not only because their gold colour is preferred, but also because they have extremely high chemical stability in the mouth, plus several desirable mechanical properties such as high strength, ductility and elasticity [1]. They contain five or more elements, the essential components of the alloys being gold and platinum.

The main specific requirements for those dental alloys used for porcelain fused to metal (PFM) technique are: a) tensile strength about 600 N/mm^2 and yield strength about 550 N/mm^2, b) hardness about 180 HV, c) elongation about 10%, d) good wetting of alloy by the porcelain and e) good biocompatibility. Prosthodontic restorations remain in an oral environment for many years and they are exposed to the corrosive influences of saliva, temperature changes, pH changes, etc. All facts mentioned above generally have an influence on the properties and biocompatibility of dental alloy. Excellent biocompatibility of Au-Pt-Zn-based alloys is obtained through the inclusion of high contents of two noble metal elements (about 96,8 wt.%).

Among various types of alloys for PMF restorations, Au-Pt-Zn-based high noble alloys have had the advantage of being around for some considerable time. They are part of clinical experience and are extremely successful [2]. The bond between the ceramic and the metal, in particular, is very strong and highly reliable [3]. With the chosen combination of the following alloying elements: Au, Pt, Zn and traces of: Ir, In, Rh, all the favourable properties of gold have been kept and its inadequate strength and hardness by Pt, Zn, Rh and Ir alloying [4] improved. When considering the formulations of Au-Pt-Zn-based high noble alloys for porcelain bonding, high Au contents are required to ensure

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biocompatibility and large Pt concentrations are necessary to sufficiently raise the melting range above the porcelain firing temperature to prevent distortion during porcelain application [2,3]. By the addition of Zn we wanted to lower the surface tension of the liquid alloy, thus enabling the material to be cast into very thin sections. The zinc also serves as a dezoxidant, together with Ir and In. Other micro-alloying metals are also added to form a thin oxide film at the surface of the alloy during the porcelain firing cycle [5, 6]. Pt, Zn and Rh are also elements which have an important role in solid solution strengthening and they are, consequently responsible for any improvement of the alloy's mechanical properties.

This alloy has no Ag which easily reacts with oxygen, gives porous castings, forms sulphur compounds, and can lead to tarnishing and ceramic discoloration (greening). This Au alloy also has no palladium or copper. Pd reacts with hydrogen; it gives porous castings and can cause allergic reactions. Cu gives reddish colour to an alloy, it reacts with oxygen and forms copper-oxide on the alloy's surface, which can cause ceramic discoloration. These are the reasons for the selected chemical composition of the alloy: about 86,9 wt.% Au, 9,9 wt.% Pt, 1,5 wt.% Zn, and 1,5 wt.% of different micro-alloying elements (Ir, In < 0,5 wt.%, Rh). Micro-alloying elements are necessary for precipitation hardening; small additions of iridium can reduce the grain size of the alloy, while the presence of rhodium enhances both strength and colour Š4Ć. For these reasons Au-Pt-Zn alloys may be attractive to patients who are allergic to palladium, copper and silver.

The aim of this work was the testing of an Au dental alloy based on the Au-Pt-Zn ternary system with minor additions of Rh, In and Ir. Emphasis was placed on the effects of different mechanical treatments on the mechanical properties and hardness of the alloy. The optimisation of proper heat treatment for Au-Pt-Zn alloy was carried-out during the first study-step. In the second study-step the microstructure of the Au-Pt-Zn dental alloy with optimal properties were determined by different characterisation techniques: optical and scanning electron microscopy in addition to EDX – chemical, ICP–OES, XRD and STA analyses.

EXPERIMENTAL WORK

The melting of very pure components (Au = 99,99 wt.%, Pt = 99,9 wt.%, Zn = 99,99 wt.%, In = 99,99 wt.%, Rh in the form of PtRh (90:10) = 99,99 wt.%, Pt in the form of PtIr (90:10) = 99,99 wt.%) were performed at Zlatarna Celje d.d. in a vacuum-induction melting furnace at vacuum p = 10^{-2} mbar and temperature T= 1873 K. Casting of the melted alloy was performed under argon pressure above 1,03 bar in a metal cast with diameter 10 mm [7]. The alloy ingot was followed by subsequent thermo-mechanical treatment (procedures of profile and polish milling, thermal treatment), and cutting-off the strip to form a regular shape. Alloy specimens were first annealed at 1223 K for 30 minutes and quenched using water. The annealed samples were then subjected to heat treatments which were carried-out in a tubular furnace (Figure 1) at selected temperatures (673, 723 and 773 K) for different times (10, 20 and 30 minutes), and finally, slowly cooled down to room temperature.

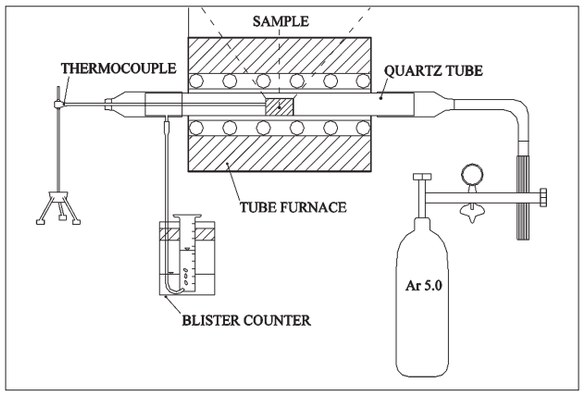


Figure 1. Schematic presentation of appliance for heat treatment.

Testing of the Au dental alloy included examining the annealed and heat treated condition. Measurements of hardness were carried-out according to standard 6507-1:1998, with the Vickers test on the Zwick 3212 microhardness measurement device. Applied load F = 49 N, according to standard was used for testing the samples. 12 measurements on the polished surface were performed for every sample. Finally the single phase' hardness of the alloy with the best previous received average value was measured.

Static tensile testing using Zwick/Roell ZO 10 tensile device was performed for determining the mechanical properties. Measurements of mechanical properties for all states were performed in one series covering 6 samples. The research conditions, as well as the shapes and dimensions of the tensile test tubes, were according to standard SIST EN 1562:2000 (chapter 6.2). Tests were performed under constant speed of increasing deformation v = 1,5 mm/min. Tensile test tubes were cast and then cut-out from the cylinder-shaped casting (diameter 3 mm).

For microscopic analysis, dental alloy plates were ground and then polished using different polish pastes. The samples were, after metallographic preparation, cleaned in an ultrasound vibration cleaning device (medium alcohol). Those specimens for optical microscopy were then etched in H_2O-H_2O_2-$FeCl_3$ solution for 2 minutes at room temperature. Concurrently those polished specimens for electron microscopy were placed in the chamber of the Sirion NC 400 high-resolution scanning electron microscope with field emission gun (FEG), having a vacuum of 10^{-10} mbar. Microscopic analysis in-

cluded examination of the polished surface and qualitative and quantitative micro-chemical analyses at characteristic points on the dental alloy. The specimens were only polished during EDX – chemical analyses in order to exclude any possible influence of chemical composition changes on the dental alloy's surface (etching medium, etc.).

ICP – OES measurements were done for the determination of chemical alloy composition. A sequence of standard solutions for obtaining the calibration plots followed, by measuring the prepared sample solutions (triplicates). The contents of the elements in the sample solutions were calculated from the obtained calibration plots.

XRD analysis was carried-out to determine the phase compositions of the Au-Pt-Zn alloy. The diffraction patterns of both alloys were collected on a PANalytical X'pert PRO MPD diffractometer using reflection geometry and $CuK_{\alpha 1}$ radiation. The 2-theta range was from 30 ° to 120 °. Diffraction patterns were compared using the X'Pert HighScore (PANalytical) computer program. The Crystallographica Search Match (Oxford Cryosystems) program was applied for qualitative phase analysis. The database used by the program was PDF-4 [8]. Crystallographic data of the identified phases were retrieved from the ICSD database [9, 10]. Quantitative phase analysis was performed using the Topas (Bruker AXS) program, which uses the Rietveld method [11,12].

Simultaneous thermal analysis of the starting materials' Au-Pt-Zn specimens was performed on a STA 449 NETZSCH machine. The measurements were carried-out in a protective atmosphere of inert gas 99.995 % Argon. The specimens were heated up to 1573 K with a heating rate of 10 K/min, isothermally kept at 1573 K for 10 min and cooled at the same cooling rate to room temperature.

RESULTS AND DISCUSSION

The average results for the hardness measurement are presented in Table 1 for each alloy state. For the annealed specimen, the average hardness value was about 125 HV. It can be concluded that the annealed alloy state has very low hardness. On the other hand there were different heat-treated (HT) samples where hardness began to increase and reached maximum value at isothermal holding by 723 K for both 20 and 30 minutes.

The average results for the mechanical properties' measurements (yield strength at 0,2 % strain, tensile strength and elongation) for tensile test tubes after different heat treatments are gathered in Table 2. With respect to the results for hardness, we can conclude that this optimal mechanical property also applied to the samples of Au dental alloy which had been heat-treated at 723 K for 20 minutes. These samples fulfil, after heat treatment, all the necessary standards regarding mechanical properties and hardness.

Table 1. **Average results for hardness measurments for Au-Pt-Zn alloy after different heat treatment / HV5.**

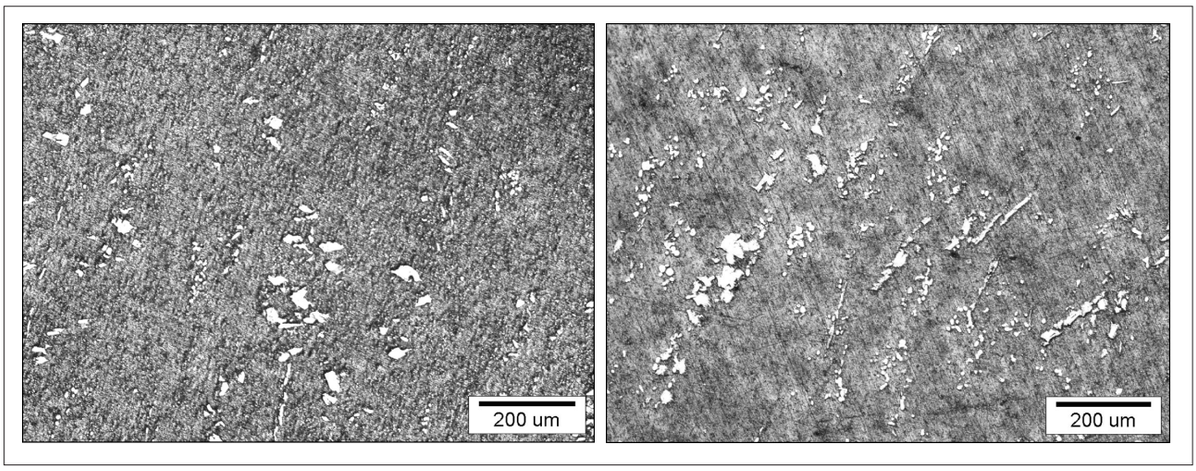
	Hardness / HV5
Initial - annealed	125
HT at 673 K, 10 min	145
HT at 673 K, 20 min	170
HT at 673 K, 30 min	175
HT at 723 K, 10 min	160
HT at 723 K, 20 min	180
HT at 723 K, 30 min	180
HT at 773 K, 10 min	160
HT at 773 K, 20 min	168
HT at 773 K, 30 min	170

The results of the ICP – OES chemical analysis of Au-Pt-Zn dental alloy (before annealing) are present in Table 3 in wt. %. This method has error ± 3 which, consequently, has an influence on the final results, especially on the essential component of the alloy - gold. For this reason, the measured value of the wt. % at Au is a little lower (approximately 0,4 wt.%).

Detailed optical microstructural examination of the annealed (Figure 2a) and heat-treated (Figure 2b) Au-Pt-Zn dental alloys with optimal mechanical properties (Au-Pt-Zn-OP) reveal that its microstructure contains two different phases: the base matrix which is identified as an Au-rich α_1-phase, and an other phase, which appears to be small particles and is known as a Pt-rich α_2 -phase [6]. The grain sizes of the α_2 phases are large as, in reality (approximately between 2-8 μm); it is a consequence of the sample's metallographic preparation (especially the influence of the etching process). Optical microscopy shows that the α_1-phase has approximately equal sizes in the annealed and heat-treated states, which

Table 2 . **The average results of mechanical properties Au-Pt-Zn alloy after different heat treatment states.**

	$R_{p0.2}$ / N/mm^2	R_m / N/mm^2	A / %
Initial - annealed	340	390	15
HT at 673 K, 10 min	450	520	9
HT at 673 K, 20 min	470	530	13
HT at 673 K, 30 min	500	560	12
HT at 723 K, 10 min	520	580	10
HT at 723 K, 20 min	550	610	9
HT at 723 K, 30 min	550	610	10
HT at 773 K, 10 min	520	570	10
HT at 773 K, 20 min	540	600	10
HT at 773 K, 30 min	550	600	13



a) b)

Figure 2. The optical microstructure of Au-Pt-Zn alloy after: a) solution treatment at 1223 K for 30 min and water quenched, b) heat treatment at 723 K for 20 min and slowly cooled to the T_{room}.

Table 3. **The results of the ICP-OES chemical analysis for Au-Pt-Zn dental alloy.**

Element / Wt. %	
Zn	1,5
Rh	0,3
In	0,1
Ir	0,3
Pt	9,9
Au	86,5

is different from α_2-phase. The size and density of α_2-phase namely change according to the condition of the heat treatment (temperature and time). Generally the higher temperature of the heat treatment leads to larger particles of α_2 -phase. The growth of α_2 –phase is in parabolic function of time which means that, after some time, particle growth stops. Examinations showed that the microstructure became homogeneous with the performing of heat treatment. α_2-phase (in the form of small particles) is located at the grain boundaries and within the grains. We envisage that the optimal and most homogeneous distribution and size of α_2-phase particles was achieved by the Au-Pt-Zn-OP sample which was heat treated at T =723 K for t =20 min after solution treatment.

At least 21 micro-hardness tests were carried-out at each phase of the Au-Pt-Zn-OP. The average results for the micro-hardness measurment of the α_2-phase show a value of about 340 HV, while the α_1-phase has about 90 HV (Figure 3).

Further phase assumptions were confirmed by EDX analysis. The gained average EDX results on the Au-Pt-Zn-OP samples show that the α_1-phase has about 87,32 wt.% Au, 10,58 wt.% Pt, 1,00 wt.% Zn; Ir, In and Rh are traceable (< 1 wt.%). On the other hand, the α_2 -phase in this sample contains about 75,64 wt.% Pt, 19,62 wt.% Au, 4,2 wt.% Rh, In and Zn are traceable, while in this phase no Ir was detected.

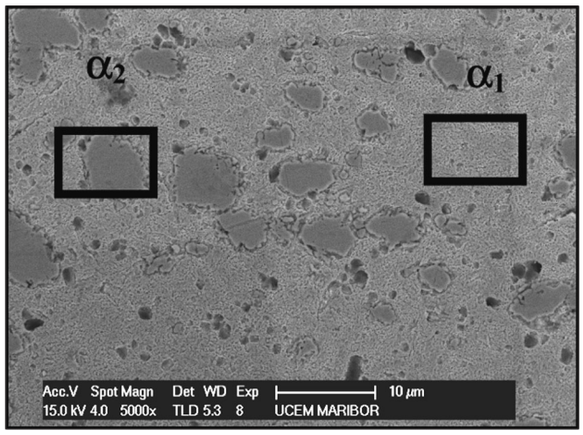


Figure 3. Electron microscopy and the typical places for micro-hardness measurements and for EDX analysis for the Au-Pt-Zn-OP alloy.

An X-ray powder diffraction curve with a combination of the Rietveld plot for the Au-Pt-Zn-OP alloy is shown in Figure 4. Measured diffraction pattern (circles) is compared to the calculated one (thin solid line), which is composed of the two components: α_1 (major) and α_1 (minor). The contribution of the latter is shown with the solid thick line at the bottom.

In addition the main phase, the Au-Pt-Zn-OP alloy contains only one minor phase (its characteristic peaks are visible, for example, at diffraction angles of 39,7, 46,3, 67,1 degrees). Qualitative phase analysis of the Au-Pt-Zn-OP alloy revealed that the major α_1-phase is similar to gold (PDF card 4-784 for pure gold specifies a cubic unit cell with a parameter of 4,0778 Å); minor α_2-phase is similar to platinum (PDF card 4-802 specifies a cubic unit cell with the parameter of 3,9231 Å).

The mass ratio of the two phases was determined by the Rietveld method. In the Au-Pt-Zn-OP alloy, there is about 98,5 wt.% of the major phase α_1 and about 1,5 wt.% of the minor phase α_2. Rietveld method also enables the determination of the unit cell parameters. The

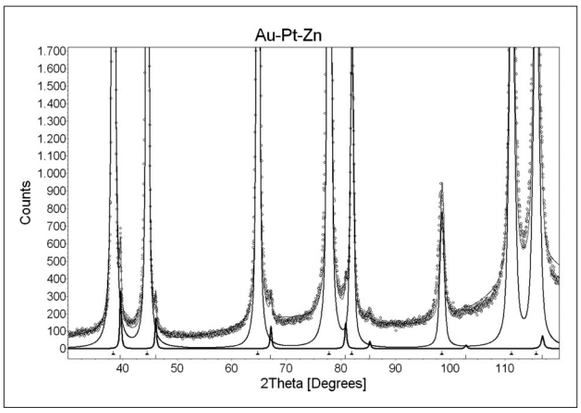


Figure 4. Rietveld plot for the Au-Pt-Zn-OP alloy.

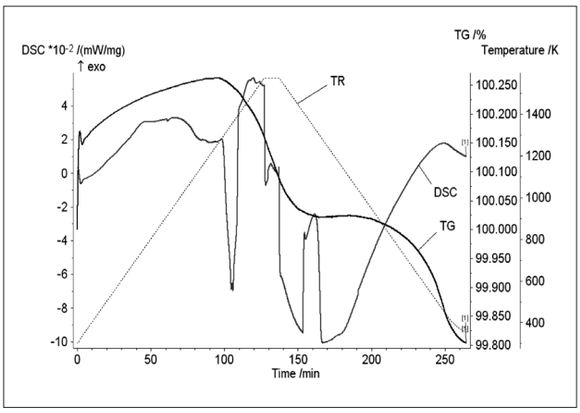


Figure 5. Results of the Au-Pt-Zn-OP alloy's STA analysis.

unit cell parameter of phase α_1 is 4,068 Å, while the unit cell parameter of the α_2 phase is 3,941 Å. Beside the peaks belonging to these two phases, there is no additional peak present in the pattern of the Au-Pt-Zn-OP alloy (Figure 4).

Figure 5 represents the results of the STA analysis: TR- demonstrates the temperature regime during the experiment, DSC represents differential scanning calorimetry cooling curve and the TG represents thermo-gravimetric analysis of the Au-Pt-Zn-OP alloy. Detailed analysis of the DSC cooling curve is in the next section. The TG curve shows the mass change in the Au-Pt-Zn-OP alloy during the STA experiment an it represents the small increment of the alloy mass 100,25 % in the heating region over a time of 100 minutes after the STA experiment's started. After that it could be observed that the alloy mass continuously falls over the isothermal holding and cooling region. The final mass balance is 99,80 % compared to the initial mass of the Au-Pt-Zn-OP alloy (100 %). We presume that the mass minus is connected to the oxidation of the Zn, In or Ir.

In Figure 6 and 7 there are DSC diagrams of the heating and cooling curves of the Au-Pt-Zn-OP alloy.

An exothermic peak in the temperature region between 453 K and 1173 K can be observed on the heating curve (Figure 6). This represents the settled solid solution (the Au-Pt-Zn-OP alloy goes into equilibrium state, consequently the heat release). Further the endothermic peak occurs at 1292,3 K, which represents the start of melting (solidus temperature). After this, two small endothermic peaks appear. We envisage that both peaks are connected with melting of phases. The first peak could be attributed to the α_1-phase and the second to the α_2-phase. 1430 K is the point where the alloy completely enters into the liquid region (L).

The solidification of the Au-Pt-Zn-OP alloy can be seen on the cooling curve (Figure 7). It starts at 1412 K. This point probably represents the liquidus temperature of the alloy. The precipitation of α_2-phase particles from the liquid begins at this point. Further appear two exothermic peaks. The small peak could be attributed to the

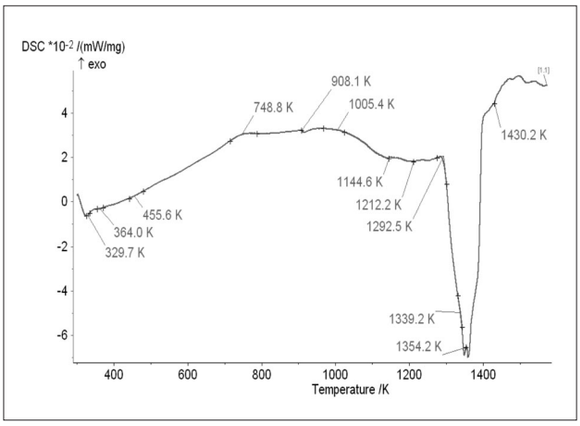


Figure 6. DSC heating curve for Au-Pt-Zn-OP alloy.

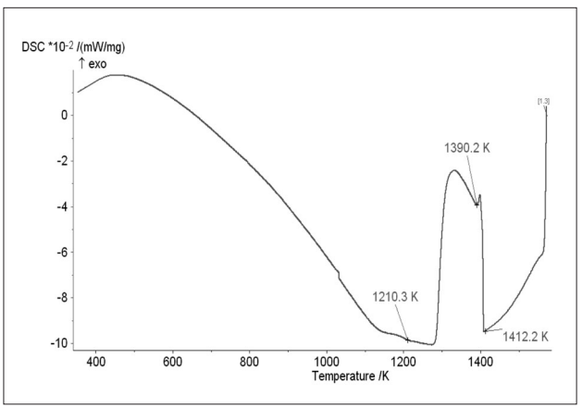


Figure 7. DSC cooling curve for Au-Pt-Zn-OP alloy.

solidification of α_2-phase and the greater attachment to the α_1-phase. The precipitation of α_1-phase probably begins at 1390 K. The surface under the exothermic peak of DSC curve and extrapolation of these regions could represent the mass portion of each phase. The determined DSC cooling curve clearly show that the mass portion of α_1-phase is much larger than the portion of α_2-phase. These results indirectly confirm the obtained results of XRD analysis where the calculated phase mass ratio is α_1:α_2 = 98,5: 1,5. In the last region at ap-

proximately 1073 K of the DSC cooling curve, precipitation in solid state occurs which can also be seen on the curve as an exothermic peak. The precipitation is connected with the formation of low temperature phase formation. We concluded that the volume portions of these phases are below 0,1 wt.%, and they could be neglected.

The present studies of heat treatment's influence on the mechanical properties of a newly- produced Au-Pt-Zn alloy show improvement in hardness, yield strength at 0,2% strain, and tensile strength during processing. These results indicate that the specimen which was heat-treated for 20 minutes at 723 K after solid solution treatment had maximum hardness and tensile strength. The reasons for these conclusions are the chemical composition of the Au-Pt-Zn alloy, microstructure formation and, consequently, the strengthening effect which occurred during heat treatment.

The Au-Pt-Zn alloy is characterized by the unknown ternary Au-Pt-Zn system. According to the obtained results and the revealed microstructure with known α_1 and α_2 phase contents the most important marginal phase diagram.

The Au-Pt binary phase diagram contains a miscibility gap (Au) + (Pt), the critical point of which is about 61 at.% Pt and at 1533 K. Miscibility gap occurs in the solid solution field, as well as the controversial form of the liquidus and solidus boundaries. The immiscibility is well-known for the Au-Pt system in a solid state. After casting, a solid solution nucleates from the melt. During cooling the alloy enters the miscibility gap separating into Au-rich and Pt-rich phases. With heat treatment at 1223 K the alloy enters the single phase zone between the miscibility gap and the solidus line [13]. At this temperature, a solid solution is thermodynamically stable with random distribution of the atoms within the crystal lattice [6]. The inhomogeneous texture formed after casting, therefore, is thermodynamically unstable. Owing to high energy, the atoms could diffuse in the crystal lattice and be forced to distribute randomly on the atomic sites. Subsequently, the alloy became homogeneous. Then during the cooling process, the homogenized microstructure kept in balance. But a small amount of the Pt-rich phase could still precipitate from the solid solution, depending on the cooling rate. Thus, all the specimens showed a microstructure composed of, not only α_1-phase, but some α_2-phase too. In the solution treatment process at 1223 K combined with water quenching, many crystal defects remained and supersaturated solid solution was achieved. After this treatment the alloy was thermodynamically in a metastable state. When metastable, specimens were put under different heat treatment temperatures they were subjected to isothermal treatment, precipitation reactions occurred driven diffusion [14].

In the case of heat treatment at 723 K for 20 minutes the precipitation reaction for α_2 phase was optimal regarding to the their size and distribution [15]. The measured micro-hardness of single phase has namely shown that α_2 phase has 4 times higher value as the α_1 phase. In this case we could presume that the α_2 phase is reinforced elements and the α_1 phase the matrix, similar to the conditions for the composite material. We supposed the α_2 phase is the main factor responsible for strengthening Au-Pt-Zn alloys according to the researched microstructure [16,17]. Homogeneous distribution, higher density and the size of α_2 phase particles clearly lead to higher values for the hardness and mechanical properties of the Au-Pt-Zn alloy. In this case we can't forget the solution strengthening of the α_1 -phase with the elements Pt, Zn, Rh, which is consequently reflected in the higher hardness value (90 HV) compared to the pure gold ($\approx$ 60 HV) [17].

Based on the results of this study, for the practical application of the present Au-Pt-Zn alloy, it is advisable to perform heat treatment.

CONCLUSIONS

1. From the results of our examinations, we can conclude that the Au-Pt-Zn dental alloy from Zlatarna Celje fulfils all the requested standards in the sense of mechanical properties and hardness. The obtained results for this alloy are: $R_{p0.2} = 550$ N/mm^2, $R_m = 610$ N/mm^2, A = 9 %, 180 HV.

2. The analyses of different heat-treated states show that the above-mentioned mechanical properties and hardness of the Au-Pt-Zn alloy can be reached with combinations of heat treatment for 20 minutes at 723 K and slowly cooling after that, if the alloy was annealed at 1223 K for 30 min and water quenched.

3. The microstructure of the Au-Pt-Zn alloy consists of two phases: α_1-phase rich in Au and α_2-phase rich in Pt. It was found that the number and density of α_2-phase increased by the performing of heat treatment. α_2-phase is in the form of small particles (2-8 μm) and is located at the grain boundaries, and within the grains. The α_2-phase shows value about 340 HV and is the main factor for the strengthening of the Au-Pt-Zn alloy.

4. The growth of α_2 –phase is in parabolic function heat treatment time and higher temperature of HT leads to more and larger particles of α_2 -phase. The optimal and most homogeneous distribution and size of α_2-phase particles was achieved in the Au-Pt-Zn-OP sample.

5. The gained EDX results on the Au-Pt-Zn samples with the optimal mechanical properties show that the α_1-phase has approximately 87,32 wt.% Au, 10,58 wt.% Pt, 1.00 wt.% Zn; Ir, In and Rh are traceable (< 1 wt.%). On the other hand, the α_2 -phase contains about 75,64 wt.% Pt, 19.62 wt.% Au, 4,2 wt.% Rh, In and Zn are traceable, while in this phase no Ir was detected.

6. Using the XRD analysis and Rietveld method, it is calculated that in the Au-Pt-Zn-OP alloy contain about

98,5 wt.% of the major phase α_1 and about 1,5 wt.% of the minor phase α_2.

7. STA analyses show that the Au-Pt-Zn alloy has solidus temperature about 1292 K and liquidus temperature about 1412 K.

8. In the Au-Pt-Zn alloy we can't forget the solution strengthening of the α_1 -phase with the elements Pt, Zn, Rh which is, consequently, reflect in the higher hardness value (90 HV) compared to the pure gold ($\approx$ 60 HV).

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