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(54) METHOD FOR OBTAINING A COMPONENT MADE OF A PALLADIUM-INDIUM ALLOY HAVING A GOOD WORKABILITY

(57) The present disclosure concerns a method for obtaining a component made of a palladium (Pd)-indium (In) alloy comprising between 46 and 56 wt% Pd, between 44 and 54 wt% In, between 0 and 10 wt% of silver (Ag), gold (Au), platinum (Pt) or tin (Sn), or a combination of these elements, between 0 and 2 wt% of a grain refiner, between 0 and 5 wt% of aluminum (Al), and a maximum of 1.5 wt% of other elements. The method comprises the steps of providing the Pd-In alloy, and thermomechanical

processing the Pd-In alloy at a heating temperature corresponding to a value between 0.6 and 0.9 of the homologous temperature (Tm) of the Pd-In alloy. The thermomechanically processed Pd-In alloy has a bending stress measured with a three-point bending test, according to DIN EN ISO 9693 until breakage that is at least two time greater that the bending stress of as-cast the Pd-In alloy measured with the same three-point bending test.

Description

Technical domain

⁵ **[0001]** The present disclosure concerns a method for obtaining a component made of a palladium (Pd)-indium (In) alloy. The component obtained by the method has a good workability.

Related art

[0002] Pd-based alloys have wide applications in jewelry and goldsmithing for the manufacture of jewelry, household utensils, objects for domestic use, etc. Pd-based alloys can also be used in the construction of precision equipment and in electronics.

[0003] More particularly, alloys based on the intermetallic phase Pd-In show a variety of colors from yellow to pink. Such alloys can be hallmarked "Palladium 500" (or Pd500). Intermetallic Pd-In alloys are generally-brittle, and not suitable for mechanical machining.

Summary

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[0004] The present disclosure concerns a method for obtaining a component made of a Pd-In alloy comprising between 46 and 56 wt% Pd, between 44 and 54 wt% In, between 0 and 10 wt% of silver (Ag), gold (Au), copper (Cu), platinum (Pt) or tin (Sn), or a combination of these elements, between 0 and 2 wt% of a grain refiner, between 0 and 5 wt% of aluminum (Al), and a maximum of 1.5 wt% of other elements. The thermomechanically processed Pd-In alloy has a bending stress measured with a three-point bending test, according to DIN EN ISO 9693 until breakage that is at least two time greater that the bending stress of as-cast the Pd-In alloy measured with the same three-point bending test.

[0005] Here, the homologous temperature Tm has its usual meaning, i.e., it expresses the thermodynamic temperature of a material as a fraction of the thermodynamic temperature of its melting point.

[0006] With respect to what is known in the art, the method disclosed herein allows for obtaining a component that has a good workability at room temperature and at moderate load. Moreover, the obtained component shows no cracking or sample failure.

[0007] Here, the term workability can include: machining, milling, drilling, turning, screw-cutting, grinding, lapping, polishing, decorating by mechanical or laser engraving, etc.

Short description of the drawings

Exemplar embodiments of the invention are disclosed in the description and illustrated by the drawings in which:

Figs. 1a-f show SEM micrographs of metallographic cross sections of components in the as-cast condition and made of a Pd-In alloy: 1H (Fig. 1a), 2A (Fig. 1b), 2B (Fig. 1c), 2ABCD (Fig. 1d), 2E (Fig. 1e), and 2F (Fig. 1f), showing the different microstructures depending on the composition;

Figs. 2a-f show SEM micrographs of metallographic cross sections of components made of a Pd-In alloy: 1H (Fig. 2a), 2A (Fig. 2b), 2B (Fig. 2c), 2ABCD (Fig. 2d), 2E (Fig. 2e), and 2F (Fig. 2f), showing the different microstructures depending on the composition, where the components have submitted to a vacuum hot pressing step at a heating temperature of 1100°C;

Fig. 3 shows a graph reporting the hardness HV1 as a function of the Al content in the Pd-In alloy;

Fig. 4 illustrates a set-up of a bending test device; and

Fig. 5 reports values for the bending stress measured on test components by using a three-point bending test.

Examples of embodiments

[0009] In an embodiment, a component is made of a Pd-In alloy comprising between 46 and 56 wt% Pd, between 44 and 54 wt% In, between 0 and 10 wt% of silver (Ag), gold (Au), copper (Cu), platinum (Pt) or tin (Sn), or a combination of these elements, between 0 and 2 wt% of a grain refiner, between 0 and 5 wt% of aluminum (Al), and a maximum of 1.5 wt% of other elements. The other elements can comprise Al, C, Co, Hf, Mg, Ni, Sb, Ti, Y, Zn, Zr.

[0010] The grain refiner can comprise ruthenium (Ru), rhodium (Rh), rhenium (Re) or iridium (Ir), or a combination of

these elements. In an embodiment, the Pd-In alloy comprises between 0.05 and 2 wt% of the grain refiner. For the Pd-In alloy comprises between 0.05 and 2 wt% of Ru.

[0011] In another embodiment, the Pd-In alloy can comprise between 1 and 10 wt% silver (Ag).

[0012] In yet another embodiment, the Pd-In alloy can comprise between 0.1 and 5 wt% aluminum (Al).

[0013] The Pd-In alloy can further comprise between 0.05 and 2 wt% Ru and between 1 and 10 wt% Ag.

[0014] The Pd-In alloy can further comprise between 0.05 and 2 wt% Ru and between 0.1 and 5 wt% aluminum (Al).

[0015] In a further example, the Pd-In alloy can comprise between 49 and 53 wt% Pd and between 46 and 51 wt% In. Here, the ratio by weight of palladium to indium is between 1.15 and 0.95.

[0016] In a further example, the Pd-In alloy can comprise between 50 and 53 wt% Pd and between 46 and 49 wt% In. Here, the ratio by weight of palladium to indium is between 1.15 and 1.02.

[0017] In one aspect, the Pd-In alloy can comprise between 0.5 and 2 wt% Al. In another aspect, can comprise between or between 1.5 and 5 wt% Al.

[0018] In an embodiment, the Pd-In alloy comprises about 50 wt% of pure Pd. The Pd-In alloy comprising 50 wt% or pure Pd can be hallmarked "Palladium 500" or Pd500, in other word, the Pd-In alloy is a Pd500 alloy.

⁵ [0019] Also disclosed is a method for preparing the component, comprising the steps of:

providing the Pd-In alloy; and

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thermomechanical processing the Pd-In alloy at a heating temperature corresponding to a value between 0.6 and 0.9 of the homologous temperature Tm of the Pd-In alloy.

[0020] The thermomechanical processing step can comprise any suitable step of thermomechanical transformation of the Pd-In alloy. For example, the thermomechanical processing step can include performing hot pressing under vacuum (vacuum hot pressing or VHP), hot pressing under a protective gas (such as a noble gas) or of hot isostatic pressing (HIP), hot rolling, extrusion, forging, or continuous casting. More generally, the heating temperature can be between 900°C or 1000 °C and the melting point of the alloy.

[0021] The step of hot pressing can be performed at controlled deformation rate of 0.1 to 100 mm/h, typically 0.1 to 10 mm/h, and more specifically 0.5 to 2 mm/h. In one aspect the applied resulting load lies between 1 and 500 MPa, typically 10 to 100 MPa. The mechanical press can also be carried out at controlled load in the above-mentioned ranges.

[0022] The step of hot pressing can be performed in an electrically heated furnace that is evacuated prior to heating. Heating can be performed at a rate of 5 to 20 K/min up to a heating temperature between 900°C and 1200°C, or preferably between 1000°C and 1200°C. At the heating temperature, the Pd-In alloy is ductile. After the heating temperature has been reached, the Pd-In alloy is uniaxially pressed and deformed at the constant heating temperature. After deformation, the component is cooled under vacuum down to 500 °C within approximately one hour and then in free cooling until room temperature is reached.

[0023] In one aspect, the step of hot pressing can be performed at a heating temperature between 1050 and 1150°C. [0024] The step of hot pressing can be performed at constant deformation rate.

[0025] The hot-pressing step comprises extruding thew alloy by passing it through a die, or mold, such as to form a wire having a diameter between 1 and 5 mm. For example, a component having an initial diameter of about 10 mm can be extruded to yield a final diameter of about 4 mm.

[0026] In the as-cast condition, the Pd-In alloy typically has irregular grain size with a columnar structure representing up to 50% of the crystal structure. Here, the expression "as-cast condition " has its usual meaning and refers to the Pd-In alloy that has not received finishing or treatment of any kind including heat treatment or hardening treatment, after casting

[0027] The as-cast Pd-In alloy has coarse-grained and irregular microstructure with grains having different size and shape. Moreover, the microstructure at the surface differs from the microstructure the center of an as-cast Pd-In alloy. The as-cast Pd-In alloy further a significant amount of porosity significantly greater than 1 % and shows shrinkage-related porosity. The shrinkage-related porosity is caused by sections of a casting that solidify later than the surrounding sections, and do not have enough metal flow into the section to completely fill. The irregular and porous microstructure of the as-cast alloy results in poor and inhomogeneous mechanical properties of the alloy. Moreover, the as-cast Pd-In alloy has fine precipitations in primary crystals. Thus, the as-cast Pd-In alloy is brittle.

[0028] The thermomechanical processing step modifies the granular structure of the Pd-In alloy and significantly reduces the zones with columnar structure, the porosity and the crystallographic inhomogeneities. The cohesion and the mechanical strength of the thermomechanically processed Pd-In alloy are thus strongly increased.

[0029] In one aspect, the method further comprises a step of obtaining an injected component by injecting the thermomechanically processed Pd-In alloy into a die or a mold at an injection temperature that is above the heating temperature and below the liquidus temperature (homologous temperature Tm). The injected component retains the improved structure of the thermomechanically processed Pd-In alloy. Re-initiating the crystallization process in the Pd-In alloy is

avoided. The step of obtaining an injected component is particularly interesting for the manufacture of serial parts in jewelry or watchmaking.

[0030] Table 1 reports the composition of different components that were prepared by the method disclosed herein. The possible other elements contained in the Pd-In alloy at a maximum of 1.5 wt% are not reported in Table 1.

Table 1

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Pd-In alloys	Composition (wt %)				
	Pd	In	Al	Ru	
1H	49.2	50.5		0.18	
2A	50,5	48.5	0.75	0.18	
2B	51.7	46.3	1.5	0.41	
2E	51.0	48.2	0.75		
2F	52.2	46.2	1.42		
2ABCD	49.2	49.0	0.1	0.49	

[0031] Figs. 1a-f show SEM micrographs of metallographic cross sections of Pd-In alloys in the as-cast condition and made of a Pd-In alloy: 1H (Fig. 1a), 2A (Fig. 1b), 2B (Fig. 1c), 2ABCD (Fig. 1d), 2E (Fig. 1e), and 2F (Fig. 1f), showing the different microstructures depending on the composition. The different Pd-In alloys show a coarse-grained and irregular microstructure. The grain shapes are equiaxed (Figs. 1a-1c, and 1e) or columnar (Figs. 1d and 1f). Differences in the microstructure at the surface and the center of can be observed in the as-cast Pd-In alloys. Furthermore, the microstructure in the as-cast Pd-In alloys can show shrinkage porosity. Such irregular and porous structure has poor and inhomogeneous properties.

[0032] Figs. 2a-f show SEM micrographs of metallographic cross sections of the components made of Pd-In alloys that have been submitted to the vacuum hot-pressing step at a heating temperature of 1100°C. More particularly, the different microstructures are shown for the component compositions: 1H (Fig. 2a), 2A (Fig. 2b), 2B (Fig. 2c), 2ABCD (Fig. 2db), 2E (Fig. 2e), and 2F (Fig. 2e). In the absence of Ru, the components 2E and 2F have large grain size (larger than 1000 μ m). The presence of Ru in the alloy composition allows for decreasing the grain size. Components 1H, 2A, 2B and 2ABCD have grain size below 100 μ m. More particularly, grain size between 30-50 μ m was estimated for the component 2A, grain size of about 20 μ m was estimated for the component 2B, grain size of about 100 μ m was estimated for the component 1H, and grain size larger than 100 μ m was estimated for the component 2ABCD. Component 2B comprising 1.5 wt% Al and 0,41 wt% Ru has the smallest grain size. The presence of Ru-precipitates in the Pd-In alloy inhibit grain growth.

[0033] The grain size of the components listed in Table 1 were estimated using optical microscopy and scanning electron microscopy (SEM) measurements of metallographic cross sections.

[0034] After performing the hot-pressing step, the component made of the Pd-In alloy has a modified microstructure. More particularly, the Pd-In alloy is deformed by the high heating temperature hot pressing step and recrystallizes. This results in a homogenization of the microstructure of the component (Figs. 2a-f). The grains have similar size and regular shape. The columnar grains disappear, and the pores are closed such that the component has a porosity of less than about 1%. The composition of the Pd-In alloy also influences the microstructure of the component as discussed below. [0035] Vacuum hot pressing significantly closes the shrinkage pores. Hot isostatic pressing can fully close the pores [0036] The hardness and the modulus of indentation were measured for the components listed in Table 1. The hardness was measured by using the hardness HV1 that corresponds to a Vickers hardness measured with a load of 1 kg (9,81N) using the measurement method defined in ISO 6507. The modulus of indentation was measured by using instrumented indentation with 500mN load, following the test specification DIN EN ISO 14577.

[0037] Table 2 reports the hardness HV1 and modulus of indentation measured of different components that were prepared by the method whereby the components were submitted to a vacuum hot-pressing step at a heating temperature of 1100°C.

[0038] Fig. 3 shows a graph reporting the hardness HV1 as a function of the Al content in the Pd-In alloy for the ascast Pd-In alloy (squares) and for the component (circles). The measurements show that hardness HV1 increases linearly with increasing Al content for the as-cast Pd-In alloy and for the component. The hardness reaches a value of above 280 HV1 for Al contents of 1,5% or more for the as-cast Pd-In alloy and for the component. The modulus of indentation measurement remains unchanged.

Table 2

Sample	Hardr	ness (HV1)	Modulus of indentation (GPa)		
	As cast	Hot pressed	As cast	Hot pressed	
1H	204	176			
2A	224	248			
2B	281	288			
2E	230	237	109	104	
2F	272	276	108	112	
2ABCD	227	209	125	115	

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[0039] More generally, it has been found that for the component comprising between 46 and 56 wt% Pd, between 44 and 54 wt% In, and a maximum of 1.5 wt% of the other elements, after being subjected to the step of hot pressing, has non-columnar crystalline microstructure, and has a porosity of less than about 1%. The crystalline microstructure has grain size between 20 μ m and 1000 μ m.

[0040] The presence of Ag in the Pd-In alloy decreases the hardness and increases the ductility of the alloy. For example, the component comprising between 46 and 56 wt% Pd, between 44 and 54 wt% In, between 1 and 10 wt% Ag, and a maximum of 1.5 wt% of the other elements has a hardness of about 200 HV1

[0041] In contrast, the presence of AI in the Pd-In alloy increases the hardness of the alloy. For example, the Pd-In alloy comprising between 46 and 56 wt% Pd, between 44 and 54 wt% In, between 0.5 and 2 wt% AI, was shown to yield a hardness HV1 between 220 and 320 HV1.

[0042] The same component but comprising between 1.5 and 5 wt% Al has a hardness HV1 that is above 280 HV1. [0043] The increased hardness observed for the component obtained by the present method can be due to any one, or a combination, of: grain refinement (Hall-Petch-relationship), formation of a solid solution, precipitation, or/and dislocations. It should be noted that the hardness is not simply related to the grain size since solid solution, precipitates and dislocations can increase the hardness in coarse grains.

[0044] The presence of the grain refiner in the Pd-In alloy strongly decreases the grain size. Ru in the Pd-In alloy strongly decreases the grain size. The Pd-In alloy has homogeneous mechanical properties. For example, the Pd-In alloy comprising between 46 and 56 wt% Pd, between 44 and 54 wt% In, between 0.05 and 2 wt% Ru, and a maximum of 1.5 wt% of the other elements has grain size below 100 μ m. In the absence of Ru, grain size strongly increases up to 1000 μ m.

[0045] Bending test using a three-point bending test according to DIN EN ISO 9693 were performed on test components made of 50.5 wt%, Pd, 48.5 wt% In, 0.8 wt% Al and 0.2 wt% Ru. In particular, the bending test were performed for three test components as-cast and three test components after the step of hot pressing under vacuum. The step of hot pressing under vacuum comprised forming the test component in a mold at 1100°C . Fig. 4 illustrates a set-up of the bending test device 1. The test component 10 is placed on two supporting pins 20 a set distance D apart, against a support 40, and is subject to a load F concentrated at its centre (exerted on a loading pin 30). The test components 10 were prepared in form of bars of about $3 \times 3 \times 25 \text{ mm}$ and were loaded in the three-point bending test device 1 until breakage. The surface of each test component 10 was finely grounded to 1200 grain. Fig. 5 reports values for the maximum bending stress measured by using the three-point bending test according to DIN EN ISO 9693, as a function of the deflection d_{ef} of the test components when the load F is applied on the test component. The bending stress is measured for three test components as-cast (No. 1-3) and three vacuum hot-pressed test components (No. 4-6), i.e., hot-pressed test components after the step of hot pressing under vacuum.

[0046] Table 3 reports the maximum bending force and maximum bending stress measured with the three-point bending test according to DIN EN ISO 9693 for three test components as-cast (No. 1-3) and three vacuum hot-pressed test components (No. 4-6). Table 3 shows that the maximum bending force and the maximum bending stress measured for the vacuum hot-pressed test components increase about three to four times compared to the maximum bending force and the maximum bending stress measured for the test components as-cast.

Table 3

No	maximum bending force (N)	maximum bending stress (MPa)	cross-sectional area of test component		
1	137	13.4	9.80		

(continued)

No	maximum bending force (N)	maximum bending stress (MPa)	cross-sectional area of test component
2	264	28.5	8.81
3	162	17.4	9.33
4	679	70.4	9.21
5	687	73.6	9.30
6	869	93.4	9.30

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[0047] When the alloy of the vacuum hot-pressed test component comprises 0.2 wt% Ru, the grains have homogeneous size and distribution, and are non-columnar and their size varies between 100 μ m and 500 μ m. When the alloy of the vacuum hot-pressed test component comprises 0.5 wt% Ru, the grains have a size of 50 μ m and without Ru the size was up to 1 mm.

[0048] The test components made of 50.5 wt%, Pd, 48.5 wt% In, 0.8 wt% Al and 0.2 wt% Ru corresponds to a hardened alloy, but embrittled by the presence of aluminum. In the absence of aluminum, the improvement in bending strength is superior because the alloy is less brittle, less hard and more ductile.

[0049] More generally, it was found that performing the thermomechanical processing step on the Pd-In alloy, it is possible to obtain a component (made of the thermomechanically processed Pd-In alloy) that has bending stress that is at least two time greater that the bending stress of the Pd-In alloy in the as-cast condition. Here, the bending stress is measured with the three-point bending test according to DIN EN ISO 9693 until breakage. The thermomechanically processed Pd-In alloy has a non-columnar crystalline microstructure and has a porosity of less than about 1%.

[0050] Color properties of the component were measured by using the CIE Standard Illuminant D65 and a 10° observer (hereafter D65/10) on metallographically polished sample of the component. In the L*a*b* color space, L stands for lightness, a* is the green / red axis, and b* is the blue / yellow axis.

[0051] The color difference (Delta E or Δ E) is the difference between two colors designated as two points in the Lab color space. A Δ E value smaller than 0.5 corresponds to almost imperceptible difference, a Δ E value between 1 and 2 corresponds to a slight color difference, a Δ E value between 2 and 4 corresponds to perceived color difference, while a Δ E value greater than 5 corresponds a different color. **Table 4** reports the color values measured for the different components reported in Table 1. The Δ E value is determined by reference with component 1H.

Table 4

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Sample	Color values					
	L*	a*	b*	ΔΕ		
1H	73,86	9,84	10,42			
2A	74.04	9.73	8.77	1.66		
2B	74.88	9.49	9.63	1.33		
2E	74.46	9.73	9.17	1.39		
2F	74.77	9.51	10.43	0.96		
2ABCD						

[0052] The component can have a color in the CIELAB color space of L* between 73 and 75, a* between 9.4 and 9.8, and b* between 9 and 10.5.

[0053] The component obtained by the method has a good workability at room temperature and at moderate load. The component shows no cracking or sample failure. Moreover, the component shows no oxidation.

[0054] In an embodiment, the component obtained by the method can constitute, completely or in part, a watch part, such as a case, a dial, a plate, a bridge, an oscillating weight, etc.

[0055] The component obtained by the method can further constitute, completely or in part, a piece of jewelry, such as a piercing, an earring, a finger-ring, a bracelet, a wrist-watch link, etc. The component can further constitute, completely or in part, a decorative component, such as a brooch, a medal, a badge, etc.

[0056] The component can further constitute, completely or in part, a component for medical applications.

Claims

1. A method for obtaining a component made of a palladium (Pd)-indium (In) alloy comprising between 46 and 56 wt% Pd, between 44 and 54 wt% In, between 0 and 10 wt% of silver (Ag), gold (Au), copper (Cu), platinum (Pt) or tin (Sn), or a combination of these elements, between 0 and 2 wt% of a grain refiner, between 0 and 5 wt% of aluminum (Al), and a maximum of 1.5 wt% of other elements;

characterized in that the method comprises:

providing the Pd-In alloy;

thermomechanical processing the Pd-In alloy at a heating temperature corresponding to a value between 0.6 and 0.9 of the homologous temperature (Tm) of the Pd-In alloy;

such that the thermomechanically processed Pd-In alloy has a bending stress measured with a three-point bending test, according to DIN EN ISO 9693 until breakage that is at least two time greater that the bending stress of as-cast the Pd-In alloy measured with the same three-point bending test.

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- 2. The method according to claim 1, wherein the grain refiner comprises any one of: ruthenium (Ru), rhodium (Rh), rhenium (Re) or iridium (Ir).
- 3. The component according to claim 1 or 2, wherein the Pd-In alloy comprises between 1 and 10 wt% silver (Ag) or between 0.1 and 5 wt% aluminum (AI).
- **4.** The method according to any one of claims 1 to 3, wherein the Pd-In alloy comprises between 0.5 and 2 wt% Al and has a hardness HV1 between 220 and 320 HV1, or between 1.5 and 5 wt% Al and has a hardness HV1 above 280 HV1.

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- 5. The method according to any one of claims 2 to 4, wherein the Pd-In alloy comprises between 0.1% and 2% wt% Ru; wherein the Pd-In alloy has grain size between 20 μ m and 100 μ m.
- 30 6. The method according to any one of claims 2 to 5, wherein the Pd-In alloy comprises between 0.1% and 0.5% wt% Ru.
 - 7. The method according to any one of claims 1 to 6, wherein the Pd-In alloy has a color in the CIELAB color space of L* between 73 and 75, a* between 9.4 and 9.8, and b* between 9 and 10.5.
 - **8.** The method according to any one of claims 1 to 7, wherein the Pd-In alloy is a Pd500 alloy.
- **9.** The method according to any one of claims 1 to 8, wherein the thermomechanical processing step comprises any one of, or a combination of, a step of hot-pressing using a mechanical press, a step of forging, a step of extrusion, a step of continuous casting, or a step of hot rolling, to form the Pd-In alloy.
- 45 **10.** The method according to claim 9,

wherein the step of hot pressing comprises a step of vacuum hot pressing at a heating temperature between 1050 and 1150°C; or a step of hot isostatic pressing.

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- 11. The method according to claim 9 or 10,
 - wherein the thermomechanical processing step comprises a step of hot-pressing; and wherein the hot-pressed alloy is extruded by passing it through a die such as to form a wire having a diameter between 1 and 5 mm.
- 12. The method according to any one of claims 9 to 11,

wherein the hot-pressing step is performed at controlled deformation rate of 0.1 to 100 mm/h, typically 0.1 to 10 mm/h, and more specifically 0.5 to 2 mm/h; or

wherein the hot-pressing step is performed under controlled load between 1 and 500 MPa, typically 10 to 100 MPa.

- **13.** The method according to any one of claims 1 to 12, further comprising a step of injecting the thermomechanically processed Pd-In alloy into a die or a mold at an injection temperature that is above the heating temperature and below the homologous temperature Tm.
- **14.** A component obtained by the method according to any one of claims 1 to 13, the component being a watch component or part of a watch component.
 - **15.** A component obtained by the method according to any one of claims 1 to 13, the component being a component or part of a component for medical, for jewelry, or decorative applications.

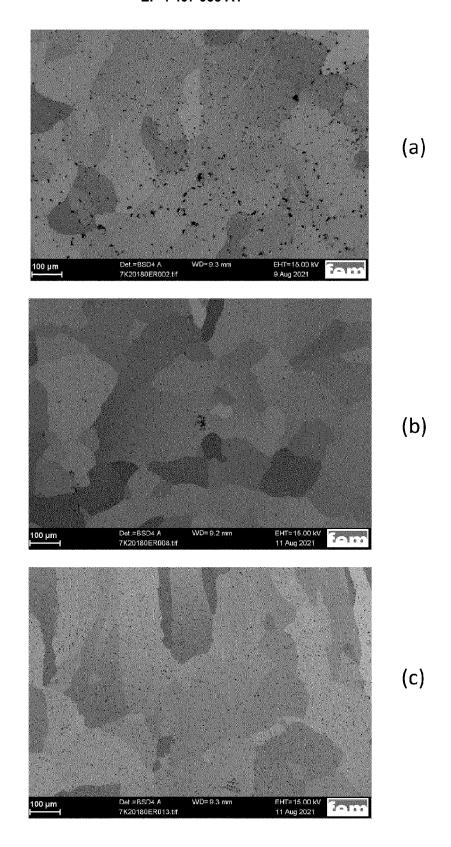


Fig. 1

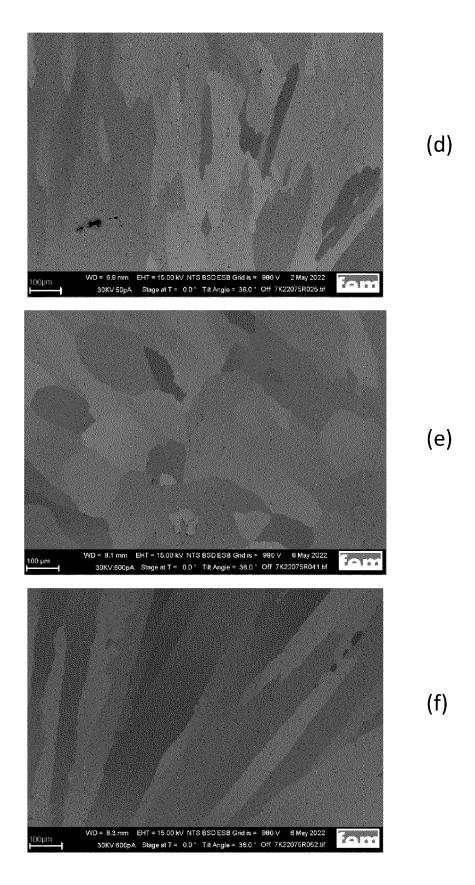


Fig. 1

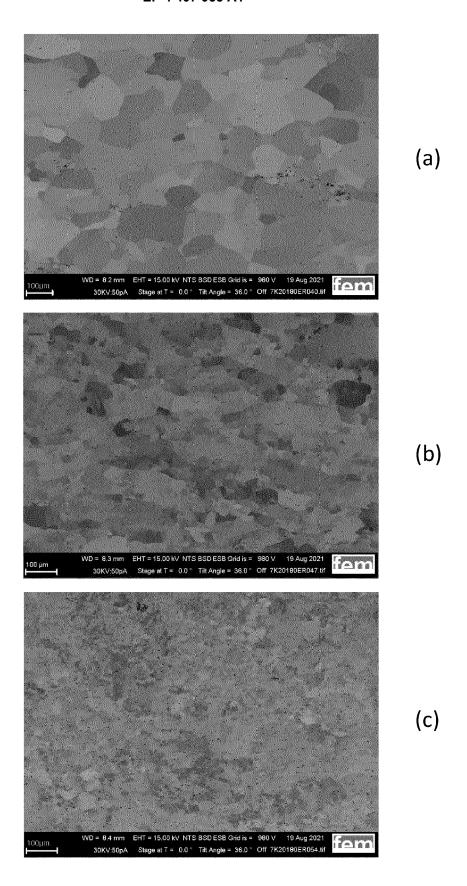


Fig. 2

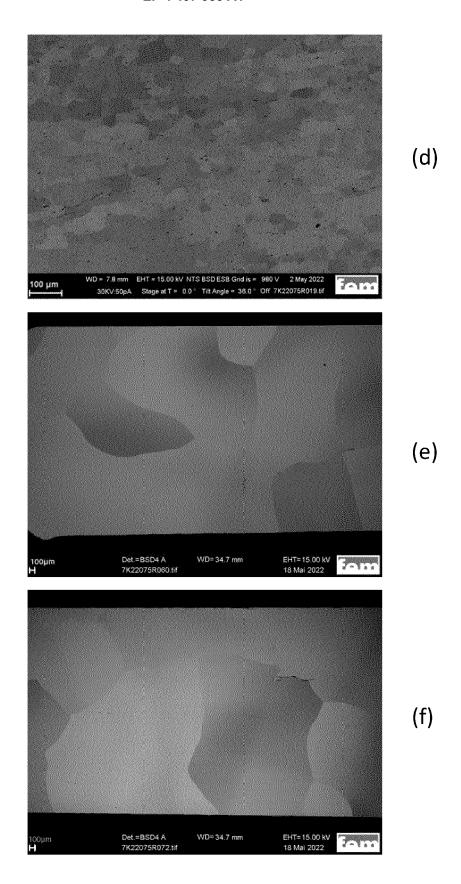


Fig. 2

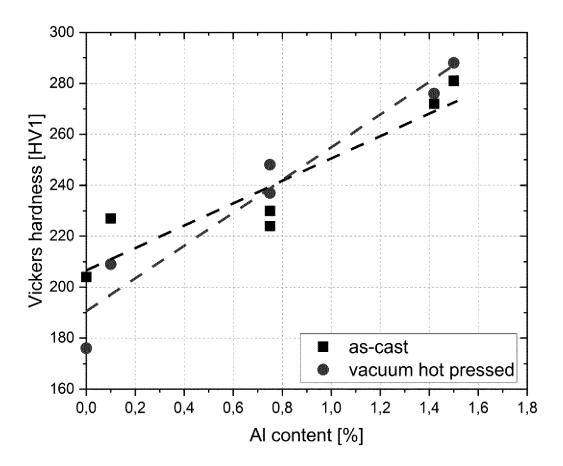


Fig. 3

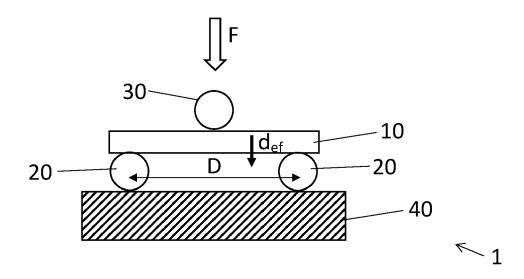


Fig. 4

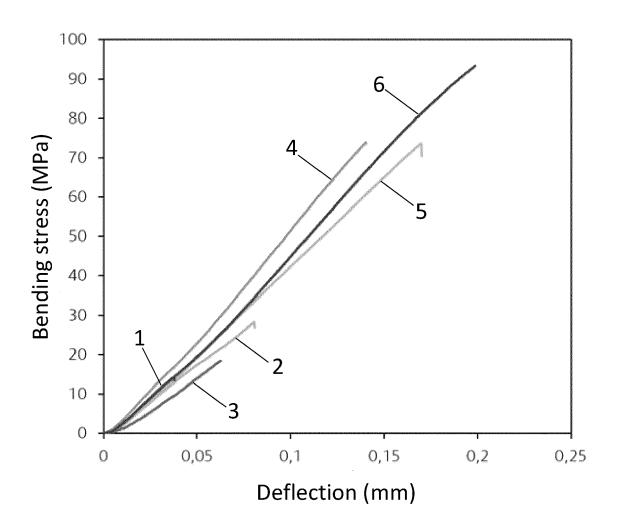


Fig. 5



EUROPEAN SEARCH REPORT

Application Number

EP 23 17 5195

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Category	Citation of document with indication of relevant passages	n, where appropriate,	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)	
x	US 4 911 762 A (STEINEM 27 March 1990 (1990-03- * column 6, lines 51-62 table 1 *	27)			
A	WO 2008/053559 A1 (LAPI: NORIYOSHI [JP]) 8 May 20 * paragraphs [0003], [1 table 1 *	008 (2008-05-08)	1-15	C22F1/02 C22F1/14	
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