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(54) **CLAY-LIKE COMPOSITION FOR NOBLE METAL SINTERED COMPACT**
TONZUSAMMENSETZUNG FÜR EINEN EDELMETALL-SINTERPRESSLING
COMPOSITION SIMILAIRE À DE L'ARGILE POUR PIÈCE FRITTÉE EN MÉTAL NOBLE

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Description

TECHNICAL FIELD

- 5 **[0001]** The present invention relates to a clay-like composition for forming a sintered precious metal body.
[0002] Priority is claimed on Japanese Patent Application No. 2013-069638, filed March 28, 2013, the content of which is incorporated herein by reference.

BACKGROUND ART

- 10 **[0003]** Conventionally, jewelry and fine arts and crafts made of precious metals, typified by rings and the like, are generally produced by the casting or forging of a material containing a precious metal such as silver or gold.
[0004] However, in recent years, precious metal clays containing precious metal powders such as silver or gold (clay-like compositions for forming sintered bodies) have become commercially available, and methods have been proposed
15 for producing precious metal jewelry and fine arts and crafts having any desired shape by making these precious metal clays into a desired arbitrary shape and then performing firing (for example, see Patent Documents 1 to 3).
[0005] By using these types of methods, precious metal clays can be used to freely shaped forms in the same manner as typical clay work. Further, by drying the shaped body obtained by shaping the precious metal clay, and then firing the clay in a heating furnace, precious metal jewelry and fine arts and crafts and the like can be produced extremely easily.
20 **[0006]** Patent Document 4 describes a thick film water based conductive composition comprising, based on total composition: (a) 5-40 wt.% first aqueous dispersion of polyhydroxyether polymer grafted with at least one acrylic or methacrylic monomer wherein the dispersion is 25-40 wt.% grafted polymer solids; (b) 20-80 wt.% finely divided metallic electrically conductive particles selected from the group consisting of silver, copper, nickel, silver coated copper, silver coated nickel, carbon, graphite and mixtures thereof; (c) 0-20 wt.% second aqueous dispersion of an acrylic or poly-
25 urethane polymer wherein the dispersion is 25-40 wt.% polymer solids; (d) 0-5 wt.% water soluble polymer; and wherein all of (a), (b), (c) and (d) are dispersed in water and about 1-8 wt.% co-solvent.
[0007] Patent Document 5 describes a method for producing a metal or alloy article which comprises mixing and kneading a metal or alloy powder with an organic binder, injection molding said kneaded mixture into a shape similar to that of the desired final product, removing the organic binder, and sintering the injected product to give a metal or alloy
30 article with high density, the organic binder comprising (a) as an adhesive agent a kind of methylcellulose having three OH groups in glucose residue contained in the cellulose, about 2 of said three OH groups being substituted with methoxy (-OCH₃) groups in a weight range of 27.5 to 31.5%, in an amount of 0.5 to 3.0% by weight, (b) as a plasticizer at least one of esters of polyhydric alcohol ether compounds, propylene glycol and polyethylene oxide in an amount of 0.5 to 3.0% by weight, (c) as a lubricant or a mold release agent at least one of a wax emulsion, a stearic acid emulsion, a
35 water-soluble acrylic resin and microcrystalline wax in an amount of 0.5 to 3.0% by weight, and (d) as a solvent for the organic binder water in an amount of 4.0 to 12% by weight, all percents by weight of the components (a) to (d) being based on the weight of the metal or alloy powder.

Citation List

- 40 Patent Documents

[0008]

- 45 Patent Document 1: Japanese Patent No. 4,265,127
Patent Document 2: Japanese Unexamined Patent Application, First Publication No. Hei 04-26707
Patent Document 3: Japanese Unexamined Patent Application, First Publication No. 2005-187858
Patent Document 4: US Patent No. 5,855,820
Patent Document 5: US Patent No. 4,721,599
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DISCLOSURE OF INVENTION

Problems to be Solved by the Invention

- 55 **[0009]** When producing a conventional sintered precious metal body mentioned above, shaping is performed using, for example, a silver clay containing a silver powder of pure Ag. This type of shaping is mainly performed by hand, but as time elapses, the clay can sometimes start to dry, making processes such as bending more difficult. Ever increasing diversity in design and personal preferences has resulted in more complex shapes for the sintered precious metal bodies,

meaning the time required for shaping these bodies has tended to increase. Specifically, with conventional materials, if an attempt is made to bend a dried silver clay, then cracks appear in the surface of the clay, and further bending can result in breakage. As a result, when rings or the like are made, the shaping must be completed within a limited time period, meaning there is a limit to the design features that are possible.

5 **[0010]** The present invention has been developed in light of these circumstances, and was completed as a result of intensive research aimed at achieving the object described below.

[0011] The present invention has an object of providing a clay-like composition for forming a sintered precious metal body which exhibits excellent flexibility and bendability during shaping of the precious metal clay, even after the precious metal clay has dried, and therefore enables the shaping time for bending and the like to be lengthened.

10 Means for Solving the Problems

[0012] In order to achieve the above object, the inventors of the present invention tested the addition of all manner of organic substances to precious metal clays (clay-like compositions for forming sintered precious metal bodies), and conducted intensive research relating to the types of additive components and the optimum amounts for those components. As a result, they discovered that by adding a prescribed amount of propylene glycol to a precious metal clay, satisfactory flexibility and bendability could be achieved during shaping of the precious metal clay, even after the precious metal clay had dried.

20 **[0013]** Moreover, as a result of further detailed testing, the inventors discovered that rather than adding only propylene glycol, also adding either one or both of glycerol and a polyethylene glycol yielded even better effects. The present invention was developed on the basis of these findings, and aspects of the invention and details relating to the development of the present invention are described below.

[0014] A clay-like composition for forming a sintered precious metal body (hereafter also referred to as a "precious metal clay") according to the present invention is obtained by adding at least an organic binder such as methyl cellulose and water to a powder containing gold, silver, or a silver alloy or the like as a precious metal, and also adding at least a prescribed amount of propylene glycol as an additive component.

25 **[0015]** The propylene glycol mentioned above is a widely-used solvent that is also used for other applications such as addition to solutions to alter the viscosity, and addition to foodstuffs or cosmetics to impart moisture retention properties. However, the effect that the addition of propylene glycol to a precious metal clay would have upon the drying of the clay during shaping was completely unknown.

[0016] In order to achieve the above object, the inventors of the present invention evaluated the addition of various types and amounts of additives to clay-like compositions for forming sintered precious metal bodies, and also evaluated the characteristics of the clay-like compositions from the viewpoints of the flexibility, bendability and formability of the compositions after drying during shaping.

35 **[0017]** As a result of continued intensive research based on the results of the above evaluations, the inventors of the present invention were able to complete the invention described below.

[0018] A clay-like composition for forming a sintered precious metal body, the clay-like composition containing at least a precious metal powder and/or a precious metal alloy powder at an amount of 50 to 95 mass%; an organic binder, an organic additive including propylene glycol, glycerol, and polyethylene glycol; and water, wherein the combined amount of the propylene glycol, the glycerol and the polyethylene glycol is 0.1 to 3.0 mass %.

Effects of the Invention

45 **[0019]** The clay-like composition for forming a sintered precious metal body according to the present invention contains at least a precious metal powder and/or a precious metal alloy powder, an organic binder, an organic additive and water, the organic additive including propylene glycol, glycerol, and polyethylene glycol, wherein the combined amount of the propylene glycol, the glycerol and the polyethylene glycol is 0.1 to 3.0 mass %, and therefore the clay-like composition exhibits excellent flexibility and bendability during shaping of the precious metal clay, even after the precious metal clay has dried. As a result, a clay-like composition for forming a sintered precious metal body can be provided which has minimal time restrictions during working of the composition by bending or the like.

BRIEF DESCRIPTION OF THE DRAWINGS

55 **[0020]**

FIG. 1 is a cross-sectional photograph of a precious metal clay according to Example 3 of the present invention, which was able to be shaped into a sheet-like form with an overall thickness of 3 mm.

FIG. 2 is a cross-sectional photograph of a precious metal clay according to Comparative Example 2, which when

stretched, partially returned to its original form, and was therefore not able to be shaped into a sheet-like form with an overall thickness of 3 mm.

BEST MODE FOR CARRYING OUT THE INVENTION

5 **[0021]** An embodiment of the clay-like composition for forming a sintered precious metal body according to the present invention is described below.

10 **[0022]** The clay-like composition according to this embodiment contains at least a precious metal powder and/or a precious metal alloy powder, an organic binder, an organic additive and water, and in particular, contains propylene glycol as the organic additive. Moreover, the clay-like composition also includes glycerol and polyethylene glycol as other organic additives, and the combined amount of the propylene glycol, the glycerol and the polyethylene glycol is from 0.1 to 3.0 mass %.

[0023] Besides the organic binder, the organic additive and water, a surface active agent, a fatty substance, and olive oil may also be added if required.

15 **[0024]** A more detailed description of the clay-like composition for forming a sintered precious metal body according to the present embodiment is described below.

(a) Precious Metal Powder, Precious Metal Alloy Powder

20 **[0025]** For the precious metal powder and/or precious metal alloy powder used in the present embodiment, gold powder, silver powder, copper powder, platinum powder, or powders of alloys of these metals can be used. Further, a mixed powder of silver powder and copper powder may also be used. In terms of silver alloy powders, a silver-copper alloy can be used particularly favorably. One or more of these precious
25 metal powders and/or precious metal alloy powders function as the main component that constitutes the clay-like composition for forming a sintered precious metal body. In order to obtain a more practical clay-like composition, the amount of the powder is at least 50mass % but not more than 95mass %. In other words, if the amount is less than 50mass %, then the texture and luster of the precious metal may not appear, whereas if the amount exceeds 95mass %, then the extensibility and strength of the clay-like composition tend to deteriorate undesirably. The amount of the precious metal powder and/or precious metal alloy powder is preferably from 75 to 93mass %.

(b) Organic Binder

30 **[0026]** The organic binder used in the present embodiment may be formed from one material, or a combination of two or more materials, selected from among cellulose-based binders, polyvinyl-based binders, acrylic-based binders, wax-based binders, resin-based binders, starch, gelatin and flour. Further, among the above, the organic binder is preferably formed from a cellulose-based binder, and most formed from preferably a water-soluble cellulose such as methyl cellulose. Although there are no particular limitations on the amount of the organic binder in the clay-like composition, the amount is typically from 5 to 50mass %, and preferably from 7 to 25mass %.

40 (c) Organic Additive

[0027] Propylene glycol is included in the organic additive used in the present embodiment. However, if the amount of this organic additive is less than 0.1mass %, then the flexibility tends to deteriorate when the clay-like composition dries, and the embodiment is unable to satisfactorily exhibit the anticipated effects. On the other hand, if the amount of the organic additive exceeds 3.0mass %, then the formability of the clay-like composition deteriorates, and shaping the composition becomes difficult.

45 **[0028]** Moreover, by also adding glycerol and/or a polyethylene glycol in addition to the propylene glycol, the flexibility of the clay-like composition after drying can be further improved.

[0029] The combined mass of the propylene glycol, the glycerol and the polyethylene glycol is adjusted to satisfy the range from 0.1 to 3.0mass %. Glycerol and polyethylene glycol are liquid substances that have both hydrophilic and lipophilic properties, and it is known that including these substances in a clay-like composition imparts viscosity to the composition. However, in the present embodiment, glycerol and a polyethylene glycol are added to the clay-like composition on the basis of the novel finding by the inventors of the present invention that by adding these substances to the clay-like composition together with propylene glycol, the effect of the invention in preventing deterioration in the flexibility of the clay-like composition upon drying can be further strengthened compared with the case where the propylene glycol is added alone.

55 **[0030]** The amount of the organic additive in the clay-like composition is more preferably from 0.5 to 2.0mass %.

[0031] Moreover, if required, at least one of a fatty substance, olive oil, and a surface active agent may also be added

to the clay-like composition for forming a sintered precious metal body according to the present embodiment.

[0032] Although there are no particular limitations on the amount of oil or fat in the clay-like composition, the amount is typically from 0.01 to 0.08mass %, and preferably from 0.03 to 0.06mass %.

[0033] Although there are no particular limitations on the amount of olive oil in the clay-like composition, the amount is typically from 0.001 to 0.05mass %, and preferably from 0.01 to 0.03mass %.

[0034] Although there are no particular limitations on the amount of the surface active agent in the clay-like composition, the amount is typically from 0.001 to 0.05mass %, and preferably from 0.01 to 0.03mass %.

[0035] Examples of the oil or fat include organic acids (such as oleic acid, stearic acid, phthalic acid, palmitic acid, sebacic acid, acetylitric acid, hydroxybenzoic acid, lauric acid, myristic acid, caproic acid, enanthic acid, butyric acid and capric acid), organic esters (such as organic acid esters having a methyl group, ethyl group, propyl group, butyl group, octyl group, hexyl group, dimethyl group, diethyl group, isopropyl group or isobutyl group), higher alcohols (such as octanol, nonanol and decanol), polyhydric alcohols (such as glycerol, arabitol and sorbitan), and ethers (such as dioctyl ether and didecyl ether).

[0036] By adding and mixing the aforementioned surface active agent into the clay-like composition, the solid matter generated by the reaction between the binder and the water can be converted to powder form, and the mixability of the precious metal powder and the binder can be improved. There are no particular limitations on the type of surface active agent, and a typical surface active agent may be used. The term "surface active agent" is a generic name for a substance that has a water-compatible portion (hydrophilic group) and an oil-compatible portion (lipophilic group, hydrophobic group) within the same molecule, and if it satisfies this definition, the aforementioned polyethylene glycol used in the present embodiment may also be considered a surface active agent. However, when used in this instance, the term "surface active agent" excludes the polyethylene glycol added with the anticipation of achieving the characteristic effects of the present embodiment, but rather refers to sodium lauryl sulfate or a polyoxyethylene alkyl ether or the like that is added in a prescribed amount in order to achieve a dispersion or aggregation effect, a foaming or defoaming effect, a wettability improvement effect, a softening and smoothing effect, or an antistatic effect or the like.

[0037] The sintered precious metal body can be produced by kneading the aforementioned clay-like composition for forming a sintered precious metal body, shaping the composition into a desired shape, and then firing the composition.

[0038] A method for producing the clay-like composition described in the present embodiment is described below. First, methyl cellulose as the organic binder, and the surface active agent, olive oil, propylene glycol, glycerol, polyethylene glycol and water are stirred and mixed inside a container fitted with a stirrer. The resulting binder mixed solution is then introduced into a kneading device together with the precious metal powder and/or the precious metal alloy powder.

[0039] In the present embodiment, the propylene glycol, glycerol and polyethylene glycol were added during the stirring and mixing of the binder mixed solution, but these components may also be added during kneading of the organic binder, the precious metal powder and/or precious metal alloy powder and water inside the kneading device.

[0040] Further, in the present embodiment, the internal walls of the stainless steel kneading container of the kneading device were coated with CrN. This CrN coating has excellent wear resistance as well as excellent lubricity, and can therefore suppress Fe contamination.

EXAMPLES

[0041] The clay-like composition of the present invention is described below in further detail using a series of examples.

[0042] First, methyl cellulose as the organic binder, and the surface active agent, olive oil, propylene glycol, glycerol, polyethylene glycol and water were stirred and mixed inside a container fitted with a stirrer to obtain a binder mixed solution. Next, this binder mixed solution was introduced, together with a silver powder, into a stainless steel kneading device having internal walls coated with CrN.

[0043] By kneading the silver powder, the binder mixed solution and water inside the kneading device, clay-like compositions having the formulations shown in Table 1 were prepared.

[0044] In the present invention examples and comparative examples described below, the description focuses on compositions in which silver powder was used as the precious metal powder, but the effect of the propylene glycol is not specific to silver powder. In other words, other precious metal powders typically used in clay-like compositions for forming sintered precious metal bodies, such as gold powder or silver alloy powders or the like, may also be used without any problems.

[Table 1]

	Component formulation of clay-like composition (mass %)						
	Silver powder	Methyl cellulose	Surface active agent	Olive oil	Propylene glycol	Glycerol	Polyethylene glycol
Reference Example 1	85	4.5	1.0	0.3	0.1	-	-
Reference Example 2	85	4.5	1.0	0.3	0.6	-	-
Reference Example 3	85	4.5	1.0	0.3	0.3	-	-
Reference Example 4	85	4.5	1.0	0.3	0.3	0.3	-
Reference Example 5	85	4.5	1.0	0.3	0.3	-	0.3
Present Invention Example 6	85	4.5	1.0	0.3	0.3	0.3	0.3
Reference Example 7	85	4.5	1.0	0.3	2.8	-	-
Comparative Example 1	85	4.5	1.0	0.3	-	-	-
Comparative Example 2	85	4.5	1.0	0.3	3.8	-	-

[Evaluation Methods]

[0045] For each of the clay-like compositions shown in Table 1, the Meyer hardness, the bendability, and the formability were evaluated.

(Meyer Hardness)

[0046] The Meyer hardness measurement is the value determined by placing a steel ball on a clay that has been dried a prescribed amount, and dividing the load applied by the cross-sectional area of the indented portion. A lower value indicates a softer material, namely a precious metal clay that exhibits greater flexibility even upon drying.

[0047] In the present invention examples and the comparative examples, measurements were performed using a sample size of $\varnothing 20$ mm with a thickness of 3 mm, and a steel ball with a mass of 140 g, namely a load [N] of 0.14×9.8 (gravitational acceleration). The results for evaluations performed at three different dried states, namely mass loss values of 0mass %, 2.0mass % and 3.2mass %, are shown in Table 2.

(Bendability)

[0048] Each of the clay-like compositions shown in Table 1 was shaped into a sheet-like form having a length of 60 mm, a width of 5 mm and a thickness of 2 mm, and following storage in a constant-temperature constant-humidity chamber at room temperature and a humidity of 40% for the storage time shown in Table 2, the sheet was rolled into a cylindrical shape of $\varnothing 17$ mm, and the occurrence of cracks on the surface of the cylinder was evaluated. The evaluation results are shown in Table 2. If no cracks of 1 mm or more appeared in the surface of the precious metal clay, an evaluation of "A" was recorded, whereas if one or more cracks of 1 mm or more appeared in the surface of the precious metal clay, an evaluation of "B" was recorded. The evaluation results are shown in Table 2.

(Formability)

[0049] A 10 g sample of each of the clay-like compositions shown in Table I was stretched on a flat plate in an attempt

to shape the precious metal clay into a sheet-like form with a thickness of 3 mm, and an evaluation was made as to whether the composition could be plastically shaped into the sheet-like form. When the composition was able to be shaped into a sheet-like form with an overall thickness of 3 mm, an evaluation of "A" was recorded, whereas when the stretched composition partially returned to its original form, meaning a sheet-like form with an overall thickness of 3 mm could not be shaped, an evaluation of "B" was recorded. FIG. 1 shows a photograph of the precious metal clay according to Reference Example 3, which was able to be shaped into a sheet-like form with an overall thickness of 3 mm, whereas FIG. 2 is a photograph showing the precious metal clay of Comparative Example 2, which when stretched, partially returned to its original form, and was not able to be shaped into a sheet-like form with an overall thickness of 3 mm. The evaluation results are shown in Table 2.

[Table 2]

	Meyer Hardness [$\times 10^{-3}$ HV]			Bendability								Formability
	Mass reduction in clay-like composition (mass %)			Storage time in constant-temperature constant-humidity chamber (hours)								
	0	2.0	3.2	0	1	12	24	48	96	144	168	
Reference Example 1	5.11	11.00	39.23	A	A	B						A
Reference Example 2	4.85	8.00	17.74	A	A	A	A	A	B			A
Reference Example 3	4.56	10.87	27.85	A	A	A	A	B				A
Reference Example 4	4.45	9.26	18.55	A	A	A	A	A	A	B		A
Reference Example 5	4.22	10.30	20.91	A	A	A	A	A	A	B		A
Present Invention Example 6	4.17	7.05	11.96	A	A	A	A	A	A	A	B	A
Reference Example 7	4.35	7.33	13.14	A	A	A	A	A	A	A	B	A
Comparative Example 1	5.32	11.20	43.69	A	B							A
Comparative Example 2	4.51	7.12	9.3	A	A	A	A	A	A	A	B	B

[0050] From the results in Table 2 it is evident that for each of the Reference Examples 1 to 5 and 7 and Invention Example 6, in which 0.1 to 3mass % of propylene glycol was added to the clay-like composition, the Meyer hardness remained lower following reduction in the mass of the clay-like composition, and flexibility was able to be maintained in the shaping operation even upon drying of the composition. Further, even after long-term storage in the constant-temperature constant-humidity chamber, the bendability did not deteriorate, and satisfactory formability was retained. In contrast, in Comparative Example 1, in which propylene glycol was not added, the Meyer hardness increased significantly following mass reduction, and the bendability deteriorated. Further, in the case of Comparative Example 2, in which 3.8mass % of propylene glycol was added, the elasticity was overly great, meaning the formability result was inferior.

[0051] Preferred examples of the present invention have been described above, but the present invention is in no way limited by these examples. Various additions, omissions, substitutions, and other modifications can be made without departing from the spirit or scope of the present invention. Accordingly, the present invention is not to be considered as being limited by the above description, and is only limited by the scope of the appended claims.

INDUSTRIAL APPLICABILITY

[0052] The present invention is able to provide a clay-like composition for forming a sintered precious metal body

which exhibits excellent flexibility and bendability during shaping of the precious metal clay, even after the precious metal clay has dried, and as a result the clay-like composition also exhibits excellent formability with minimal time restrictions during working of the composition by bending and the like.

5 [0053] Further, a sintered precious metal body can be produced by kneading the clay-like composition for forming a sintered precious metal body according to the present invention, shaping the composition into a desired shape, and then firing the composition.

10 Claims

1. A clay-like composition for forming a sintered precious metal body, the clay-like composition comprising:

at least a precious metal powder and/or a precious metal alloy powder at an amount of 50 to 95 mass%;
an organic binder;

15 an organic additive including propylene glycol, glycerol, and polyethylene glycol; and
water, wherein the combined amount of the propylene glycol, the glycerol and the polyethylene glycol is 0.1 to 3.0 mass %.

20 Patentansprüche

1. Tonartige Zusammensetzung zur Bildung eines gesinterten Edelmetallkörpers, wobei die tonartige Zusammensetzung

zumindest ein Edelmetallpulver und/oder ein Edelmetallegierungspulver in einer Menge von 50 bis 95 Masse-%;

25 ein organisches Bindemittel;

ein organisches Additiv, einschliesslich Propylenglykol, Glycerin und Polyethylenglykol; und

Wasser,

umfasst, wobei die kombinierte Menge an Propylenglykol, Glycerin und Polyethylenglykol 0,1 bis 3,0 Masse-% beträgt.

30 Revendications

1. Composition similaire à de l'argile pour former un corps fritté en métal noble, la composition similaire à de l'argile

35 comprenant :

au moins une poudre de métal noble et/ou une poudre d'alliage de métal noble en une quantité de 50 à 95 % en masse ;

un liant organique ;

40 un additif organique incluant du propylène glycol, du glycérol, et du polyéthylène glycol ; et

de l'eau, dans laquelle

la quantité combinée du propylène glycol, du glycérol, et du polyéthylène glycol est de 0,1 à 3,0 % en masse.

FIG. 1

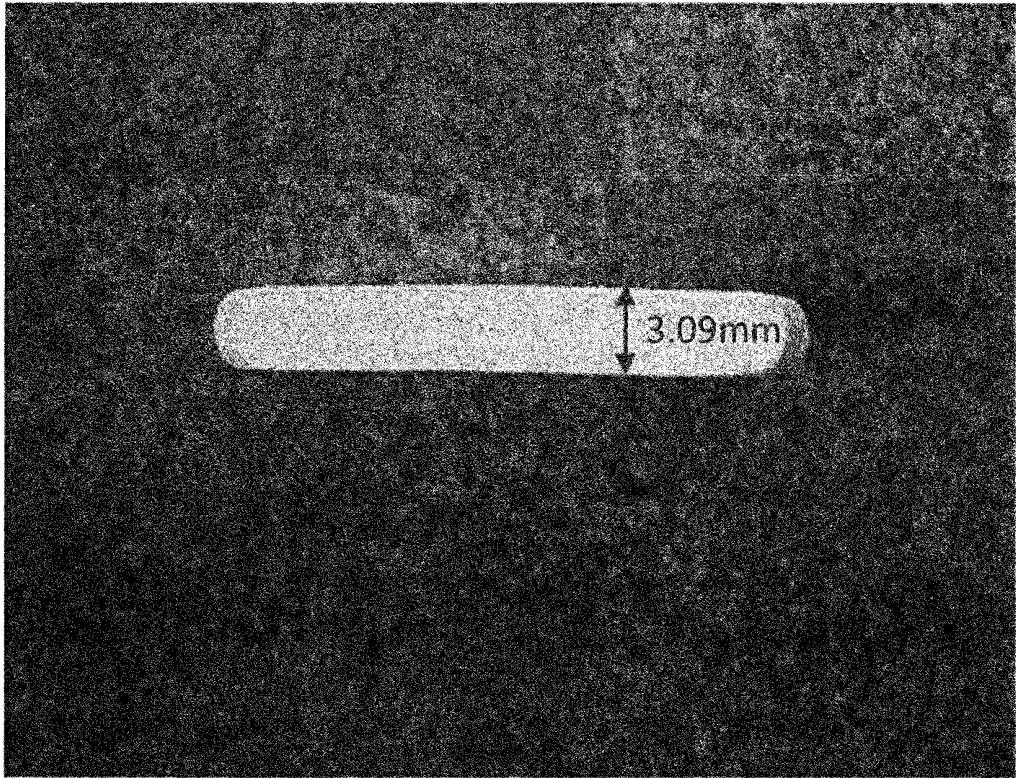
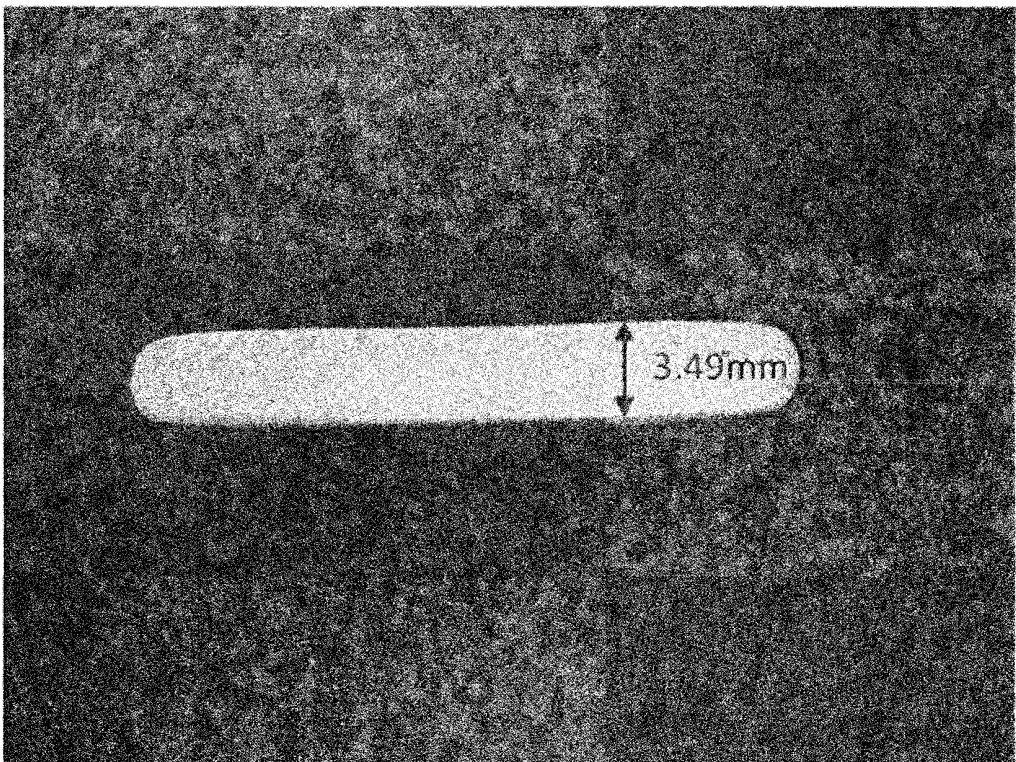


FIG. 2



REFERENCES CITED IN THE DESCRIPTION

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