Effect of Sputtering Method on Characteristics of Amorphous Ni–Nb–Zr Alloys for Glass Lenses Molding Die Materials*

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Abstract

We investigated the characteristics of amorphous Ni–Nb–Zr alloys for fabricating dies for molding high-precision optical glass lenses. Two amorphous Ni–Nb–Zr samples with similar compositions were prepared using carousel-type and parallel plate sputtering systems; we refer to these samples as the *synthesized sample* and the *target sample*, respectively. These two samples were evaluated and compared the desired characteristics for molding dies for optical glass lenses, namely a high mechanical strength, a high hardness, a thermally stability of amorphous state, a high oxidation resistance, a similar linear expansion coefficients to that of glass, and a high machinability. The thermal stability of amorphous state, oxidation resistance, and linear expansion coefficient did not depend on the fabrication method, whereas the mechanical properties, hardness, and machinability did. In particular, the target sample exhibited excellent mechanical strength and machinability. Finally, we fabricated a molding die made from amorphous Ni₃₅Nb₄₀Zr₂₅ alloy for optical glass lenses using diffraction gratings.

Key words: Amorphous Alloy, Sputtering, Optical Glass Lens Mold, Machinability

1. Introduction

Recently, functional optical glass lenses with precise microstructures, such as diffractive gratings or micro lens arrays, have been the subject of active and rapid development. Currently, optical glass devices are produced using glass molding techniques with molds made of tungsten carbide ^{(1), (2)}. However, it is difficult to fabricate precise microstructures in tungsten carbide by machining processes because of significant tool wear. Moreover, since tungsten carbide is a sintered material with microparticles of tungsten carbide and Co-binder, there is a tendency for the tungsten carbide particles to drop out at the edges of parts ⁽²⁾. Although many researchers have been investigating the fabrication of precision tungsten carbide molds for optical glass devices through improvements in the machining processes ^{(1), (2)}, this could take significant time.

In the meantime, researchers have also been searching for new candidate molding die materials to replace tungsten carbide. In particular, amorphous alloys are expected to be excellent candidate molding die materials for functional optical glass lenses with precise microstructures. Currently, amorphous alloys, such as electroless Ni-P platings ^{(3), (4)}, are being used industrially for molding die materials of optical plastic devices, such as pick-up lenses compatible with CD – DVD drives or optical elements for LCD or projection TVs, ⁽⁴⁾ because they show good machinability using diamond cutting tools ⁽⁴⁾⁻⁽⁶⁾. Some researchers, including our group, had been attempting to apply electroless Ni-P

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platings to glass lens molding dies ^{(6),(7)}. However, molding dies made of electroless Ni-P platings are destroyed during the glass molding process, and their lifetimes are too short for industrial applications. Generally, the molding temperature of glasses is more than 700 K (723 K, in the case of the commonly known glass material BK7 (Shotto AG)) ⁽⁸⁾. However, the crystallization temperature T_x of the electroless Ni-P plating is approximately 623 K, lower than the glass molding temperature ⁽⁹⁾. Thus, electroless Ni-P platings crystallize during the molding process and become brittle.

In our previous studies, we have been searching for new amorphous alloys for glass lens molding die materials using a combinatorial technique ^{(8),(10)-(12)}. So far, we have investigated the properties of Pt ^{(8),(10)} and Ni-based ^{(11),(12)} amorphous alloys for glass lens molding die materials. Amorphous Ni–Nb–Zr alloys are promising materials for fabricating molding dies for glass lenses. We used the combinatorial approach to search for suitable amorphous Ni–Nb–Zr alloys for fabricating molds for glass lenses, and we investigated the characteristics of 17 different amorphous Ni–Nb–Zr alloys ^{(11),(12)}. These samples were synthesized by carousel-type sputtering. Instead of preparing 17 different alloy targets, this method allows for the fabrication of samples with various alloy compositions by varying the radio frequency (RF) power supplied to the target of each constituent element ⁽¹³⁾. Here, we refer to these samples as *synthesized samples*. Based on the results of the previous studies, we concluded that the amorphous Ni₃₆Nb₃₉Zr₂₅ alloy is a suitable molding die material for glass lens molds because it has high thermal stability in the amorphous state, high mechanical strength, linear expansion coefficient similar to that of conventional glass, and good machinability.

Figure 1 shows a schematic of the process used for fabricating a glass lens molding die using an amorphous alloy. The amorphous alloy is sputter-deposited on the optical aspheric surface of a blank molding die. A portion of the amorphous alloy is then cut using a single-crystal-diamond cutting tool to form a precise mold pattern for an optical element such as a diffraction grating. In industrial fabrication processes, amorphous alloys are generally sputter-deposited using a conventional parallel-plate sputtering system with an alloy target, because such a system yields a reproducible alloy composition and higher sputtering rates than a carousel-type sputtering system. In this paper, we refer to these samples as *target samples*. However, there is concern that the properties of amorphous alloys are adversely affected by this fabrication method.

In this study, we compare the characteristics of the *synthesized* and *target* amorphous alloys, and demonstrate the fabrication of a molding die for glass lenses with diffraction gratings using a *target* amorphous Ni–Nb–Zr alloy.

Fig. 1 Process for fabricating a molding die for glass lenses using an amorphous alloy

2. Experimental Methods

Amorphous Ni–Nb–Zr alloys were prepared using the carousel-type RF magnetron sputtering system (L-350S-C, Cannon Anelva) shown in Fig. 2. This system has a rotating substrate holder at the center of a chamber and several targets positioned around the chamber wall ⁽¹³⁾. Two kinds of samples were fabricated using this system. Synthesized samples were fabricated by rotating the substrate holder at 50 rpm and co-sputtering several pure metal targets on a substrate. On the other hand, this system

could be used as a conventional parallel-plate sputtering system by fixing the substrate holder and using an alloy target. Target samples were fabricated by this method.

Table 1 shows the sputtering conditions for these two kinds of amorphous Ni–Nb–Zr samples. Both samples were deposited on Al₂O₃ substrates for measurements of their compositions, thicknesses, oxidation resistances, and machinabilities, and on Cu foils measurements of their mechanical strengths and linear expansion coefficients. The thicknesses of both kinds of samples were measured using a white-light interferometer (NewView 5032, Zygo) and were found to be approximately 5 μm. The compositions of both kinds of samples were measured by energy-dispersive X-ray fluorescence spectroscopy (μEDX-1300, Shimadzu). The synthesized sample had a composition of Ni₃₆Nb₃₉Zr₂₅, whereas the target sample had a composition of Ni₃₅Nb₄₀Zr₂₅ (at.%).

The mechanical properties were measured by performing tensile tests using a thermal mechanical analyzer (TMA; TMA-60, Shimadzu) with a strain rate of 0.0017 s^{−1}. Prior to the tensile tests, the Cu foils were dissolved in HNO₂ + H₂O (1:1). The samples were cut into 1 × 7 mm² strips using a diamond wire saw (Musashino Electron). Hardness was measured by the nanoindentation method using an atomic force microscope (SPI-3000, Seiko Instruments) equipped with a nanoindenter (Triboscope, Hysitron). Hardness tests were performed at five positions on each sample. Linear expansion coefficients were measured by performing thermal expansion tests using the TMA. The heating rate was 0.33 K/s. The measurement temperature was varied in the range 303 to 573 K.

Both kinds of samples on Al₂O₃ substrates were annealed at 723 K for various times in a vacuum using an image furnace (MILA-3000, Ulvac Technology) to evaluate the thermal stability of their amorphous states and in an air using a muffle kiln (MMF-1, As One) to evaluate their oxidation resistances. The phases of the annealed samples were identified by X-ray diffraction (XRD; RINT 2200, Rigaku).

The machinabilities of the samples were evaluated by performing cutting tests with an ultra-precision diamond machining system (Nanoform 350, Precitech). Figure 3 shows a schematic of the cutting test setup. The workpiece used in the tests was a sample deposited on an Al₂O₃ substrate. The workpiece was in the workpiece holder, which was rotated at 200 rpm. The workpiece was cut in a tapered shape using a single-crystal-diamond radius

Fig. 2 Carousel-type sputtering system

Table 1 Sputtering conditions for synthesized and target Ni–Nb–Zr samples

Samples	Synthesized samples	Target samples
Targets	Ni (2N up), Nb (3N), Zr (2N up)	Ni ₃₅ Nb ₄₀ Zr ₂₅ (at.%)
RF powers	Ni: 100, Nb: 267, Zr: 160 W	200 W
Sputtering times	21,600 s	2400 s
Base pressure	< 5 × 10 ^{−4} Pa	
Ar pressure	1.0 Pa	

cutting tool with a nose radius of 0.5 mm, a rake angle of 0° , and a clearance angle of 5° . The radial feed was $1 \mu\text{m}/\text{rev}$, the axial feed was $1 \text{ nm}/\text{rev}$, and the target cutting angle θ_0 (= cutting depth/cutting width) was $1 \mu\text{m}/\text{mm}$. The feed start position was 3.5 mm from the rotation center. The feed distance in the radial direction was 3 mm. The cut surface was observed using the white-light interferometer. In this study, we define the removal fraction F_r as the index of machinability, which is expressed as $\theta/\theta_0 \times 100\%$, where θ is the actual cutting angle.

Fig. 3 Set up for the cutting tests

3. Results and Discussion

3.1 Mechanical properties

Molding die materials require a high compression strength to prevent molding dies from breaking during cooling after molding. However, since the two samples in this study were too thin to evaluate their compression strengths, we measured their tensile strength instead. The fracture stress σ_f was evaluated during the tensile test; ideally, σ_f should be more than 1.0 GPa.

Figure 4 shows stress–strain curves for both Ni–Nb–Zr samples. Both samples fractured during elastic deformation. The synthesized sample had a fracture stress σ_f of 1.1 GPa, a fracture strain ε_f of 1.8%, and a Young's modulus E of 62.0 GPa, whereas the target sample had σ_f of 1.7 GPa, ε_f of 2.4%, and E of 70.9 GPa. Both samples have sufficiently high fracture stresses (over 1.0 GPa) to enable them to be used as die materials for molding glass. The target sample had a higher fracture stress and strain and Young's modulus than the synthesized sample.

Molding die materials are required to have a high hardness to ensure good mold transferability. The hardness should be 8.0 GPa or higher. The hardnesses of both samples were measured by the nanoindentation method. The synthesized and target samples had hardnesses of 8.0 and 10.0 GPa, respectively. Thus, the target sample was harder than the synthesized sample. These results demonstrate that the synthesized and target samples have different mechanical properties.

Fig. 4 Stress–strain curves of the amorphous Ni–Nb–Zr alloys

3.2 Linear expansion coefficients

Molding die materials are desired to have similar linear expansion coefficients to that of glass to prevent optical glass lenses and the molding die from breaking during cooling after molding. In this study, we assumed that BK7 is used as the glass. BK7 is a popular glass for optical components. The linear expansion coefficient of BK7 is $8.3 \times 10^{-6} / \text{K}$ for temperatures in the range 293 to 573 K⁽¹⁴⁾.

Figure 5 shows strain–temperature curves for thermal expansion tests of the thin-film amorphous Ni–Nb–Zr alloys. The initial stress of 10 MPa was applied to the samples by the weight of the bottom chuck gripping the end of the samples. Both samples expanded with increasing temperature. The mean linear expansion coefficients were estimated by the method of least squares to be $8.3 \times 10^{-6} / \text{K}$ for the synthesized sample and $7.9 \times 10^{-6} / \text{K}$ for the target sample. Both samples had similar linear expansion coefficients to that of BK7. This result demonstrates that the linear expansion coefficient does not depend on the fabrication method.

Fig. 5 Strain–temperature curves of the amorphous Ni–Nb–Zr alloys

3.3 Thermal stabilities of amorphous state and oxidation resistances

Molding die materials need to have high thermal stabilities at the molding temperature. Amorphous alloys require an amorphous state with a high thermal stability to prevent crystallization from occurring during molding because crystallization of the amorphous alloy might make the alloy brittle or form a rough surface. During molding of optical lenses made from BK7, the molding dies are heated at 723 K. Consequently, we evaluated the thermal stabilities of the amorphous states of the two alloy samples at 723 K in vacuum. The maximum annealing time was set to 100 h because the target lifetime for a molding die is 100 h; this lifetime permits 10,000 glass lenses to be fabricated from a single molding die.

Figures 6(a) and (b) show XRD profiles for synthesized $\text{Ni}_{36}\text{Nb}_{45}\text{Zr}_{25}$ samples and target $\text{Ni}_{35}\text{Nb}_{40}\text{Zr}_{25}$ samples annealed at 723 K for various times in vacuum, respectively. All the XRD profiles contain several sharp peaks corresponding to the Al_2O_3 substrate. Both annealed samples have a halo pattern due to the amorphous phase and no peaks corresponding to crystalline phases. These results indicate that both samples exhibit excellent thermal stabilities of amorphous state.

In addition, it is desirable for molding die materials to have high oxidation resistances. This is because glass lenses are fabricated in air, rather than in vacuum or inert atmospheres (e.g., N_2 or Ar) to improve the productivity of glass lenses and to reduce manufacturing costs.

Figures 7(a) and (b) show XRD profiles of the synthesized $\text{Ni}_{36}\text{Nb}_{39}\text{Zr}_{25}$ samples and the target $\text{Ni}_{35}\text{Nb}_{40}\text{Zr}_{25}$ samples annealed at 723 K for various times in air, respectively. All the XRD profiles contain several sharp peaks due to the Al_2O_3 substrate. The

Fig. 6 XRD profiles of (a) synthesized and (b) target Ni–Nb–Zr samples annealed at 723 K for various times in vacuum

Fig. 7 XRD profiles of (a) synthesized and (b) target Ni–Nb–Zr samples annealed at 723 K for various times in air

synthesized samples (Fig. 7(a)) that were annealed for less than 50 h exhibited a halo pattern, which indicates that they are in the amorphous state. However, the intensity of the amorphous peak was weaker in the sample annealed for 50 h. The sample annealed for 100 h showed two broad peaks at about 31° and 42° . We could not identify these peaks, but it is considered that they are due to metallic oxides.

The target samples (Fig. 7(b)) that were annealed for less than 50 h also showed a halo pattern. However, the intensity of the amorphous peak in the sample annealed for 50 h was weaker. The sample annealed for 100 h showed peaks due to $\text{Ni}_{11}(\text{Nb,Zr})_9$ and NbO_2 , in addition to the halo pattern.

To investigate the effect of annealing in vacuum and in air on the surface roughness, we used the white-light interferometer to measure the surface roughness R_a of the annealed samples. Figure 8 shows the surface roughnesses R_a of the annealed samples in vacuum and air. The as-sputtered target samples had an R_a of 4.8 nm, which is lower than

Fig. 8 Surface roughnesses of the annealed samples in vacuum and air

that of the Al_2O_3 substrate (6.0 nm). In contrast, the as-sputtered synthesized samples had an R_a of 7.6 nm, which is greater than that of the Al_2O_3 substrate. The different R_a of the both samples is discussed in section 3.5. The R_a of both samples annealed for 10 h in vacuum was only slightly higher than that of the corresponding as-sputtered sample. This slight increase in the surface roughness might be caused by slight surface oxidation that was not detected by XRD measurements or by structural relaxation, such as residual stress relaxation or the filling of voids. In contrast, the R_a of both samples annealed in vacuum varied little with increasing annealing time. Thus, these alloys exhibit a sufficiently high thermal stability of their amorphous states to be used as glass molding die materials. There was no detectable difference between the thermal stabilities of the amorphous states of the two samples.

On the other hand, the R_a of both samples annealed in air increased slightly for annealing times less than 20 h. The R_a of the samples annealed for more than 50 h increased further and R_a of the samples annealed for 100 h increased to 10.6 nm for synthesized samples, and 7.4 nm for target samples.

The effect of annealing time on R_a is consistent with the XRD measurements. The XRD results reveal that none of the samples annealed in vacuum were obviously crystallized or oxidized. Consequently, there was only a slight change in R_a on annealing. On the other hand, both samples annealed in air for less than 20 h showed only an amorphous peak in their XRD profiles. The R_a of these samples is the similar to that of the as-sputtered samples. For both samples, the amorphous peak intensity decreases when the annealing time is 50 h, which indicates that these samples were partially oxidized (although this was not detected by the XRD measurements). Thus, the surfaces of these samples became rough. On further annealing, the crystalline phase and/or metallic oxides appeared, making the surface rougher. These results imply that if molding dies for glass lenses are fabricated from these samples and are used in air their lifetimes might be less than 50 h.

3.4 Machinability

Molding die materials require good machinabilities so that precise microstructures, such as diffraction gratings, can be fabricated on the optical surface of the molding die using a diamond cutting tool. Figures 9(a) and (b) show the cutting profile and surface roughness of the synthesized $\text{Ni}_{36}\text{Nb}_{39}\text{Zr}_{25}$ sample, respectively. The synthesized samples could be cut into tapered shapes. The total cutting width in the radial direction was 0.88 mm and the total cutting depth in the axial direction was 0.62 μm . However, the actual cutting distance in the radial direction was shorter than the feed distance of 3 mm. This is due to an error in setting the workpiece and a slight variation in the thickness of the amorphous alloy. These factors affected the cutting test results because the target cutting angle is very small (5.73×10^{-2} deg or 1 nm/1 μm). The removal fraction F_r of the synthesized sample was 70.5% (cutting depth/cutting width: 0.62 μm /0.88 mm) and the R_a of the cutting surface was 4.0 nm.

Figures 10(a) and (b) show the cutting profile and the surface roughness of the target $\text{Ni}_{35}\text{Nb}_{40}\text{Zr}_{25}$ sample, respectively. The target samples could also be cut into tapered shapes. The total cutting width in the radial direction was 2.24 mm and the total cutting depth in the axial direction was 2.15 μm . F_r was 96.0% and R_a was 4.0 nm, which is equal to the R_a of the synthesized sample. These results reveal that the target sample has a higher F_r than the synthesized sample. The target sample exhibited excellent machinability.

3.5 Different characteristics of the synthesized and target samples

Comparing the characteristics of the synthesized and target samples reveals that both samples have similar thermal characteristics, including linear expansion coefficients,

Fig. 9 (a) Cutting profile and (b) surface roughness of synthesized $\text{Ni}_{36}\text{Nb}_{39}\text{Zr}_{25}$ sample

Fig. 10 (a) Cutting profile and (b) surface roughness of target $\text{Ni}_{35}\text{Nb}_{40}\text{Zr}_{25}$ sample

thermal stabilities of their amorphous states, and oxidation resistances. On the other hand, they have different mechanical characteristics and machinabilities. These differences are considered to be caused by the configurations of the samples.

Figures 11(a) and (b) show SEM images of the surfaces of as-sputtered synthesized and target samples, respectively. Both samples exhibited grains. These grains are several micrometers in size. They are not crystalline grains but are traces of the crystalline grains of the Al_2O_3 substrate. The synthesized sample has a rough surface with many particles, which are less than $2\text{ }\mu\text{m}$ in size. In contrast, the target sample has a smooth surface and with several dimples, which are about $0.5\text{ }\mu\text{m}$ in size. This difference in the microstructures of the samples is considered to be responsible for the different surface roughnesses of the two as-sputtered samples (Fig. 8).

The microstructure of the synthesized sample corresponds to a columnar structure originating from sputter deposition at low sputtering rates caused by using a low RF power and a high Ar pressure⁽¹⁵⁾. In our previous study of Pt-based amorphous alloys, columnar

Fig. 11 SEM images of as-sputtered (a) synthesized and (b) target samples

structures were much more obvious ⁽¹⁰⁾. However, since the average RF powers of each target (shown in Table 1) used in the present study are larger than those in the previous study (e.g., the RF powers of the synthesized $\text{Pt}_{50}\text{Zr}_{36}\text{Ni}_{14}$ sample were Pt: 100 W, Zr: 200 W, and Ni: 83 W ⁽¹⁰⁾), columnar structures were not so obvious in the synthesized $\text{Ni}_{36}\text{Nb}_{39}\text{Zr}_{25}$ sample. Since it is considered that the columnar structure is porous, the different microstructures of the two samples are expected to affect their mechanical properties more than their thermal characteristics. Thus, the synthesized sample has lower fracture stress and strain and hardness than the target sample. The difference in the Young's moduli of the two samples is caused by residual stress induced during sputtering as well as by the different microstructures, since the residual stress is affected by the sputtering conditions. Annealing has been reported to change the Young's modulus of a sputter-deposited amorphous alloy due to residual stress relaxation ⁽¹⁶⁾.

Based on the machinability results, we conjecture that the machinabilities are affected by the microstructure in micro or nanomachining. We intend to investigate the effect of the sputtering conditions and microstructures on the machinabilities of thin-film amorphous alloys in future studies.

3.6 Fabrication of molding die for glass lenses from amorphous $\text{Ni}_{35}\text{Nb}_{40}\text{Zr}_{25}$ alloy

We attempted to fabricate a molding die for glass lenses with diffraction gratings using amorphous $\text{Ni}_{35}\text{Nb}_{40}\text{Zr}_{25}$ alloy. The amorphous $\text{Ni}_{35}\text{Nb}_{40}\text{Zr}_{25}$ alloy was deposited on a blank STAVAX (Uddeholm) die that had been worked to an aspheric surface and was approximately 100 μm thick. A glass lens mold was fabricated using an ultraprecision diamond machining system. Roughness machining was performed using a single-crystal diamond tool with a nose radius of 0.01 mm, a rake angle of 0° , and a clearance angle of 5° . The workpiece was rotated at 100 rpm. The radial feed was 200 $\mu\text{m}/\text{min}$ and the cutting depth was 1.0 μm . Final machining was performed using a sword-shaped single-crystal diamond tool with a clearance angle of 10° . The workpiece was rotated at 200 rpm. The radial feed was 100 $\mu\text{m}/\text{min}$ and the cutting depth was 0.2 μm . The amorphous alloy was well adhered to the STAVAX substrate since the amorphous alloy did not peel off from the blank die during either machining process.

Figure 12(a) shows a photograph of a molding die for glass lenses and Figs. 12(b) and (c) show SEM images of the optical surface. This is molding die for a 5-mm-diameter pick-up lens. Figure 12(c) shows an SEM image of the periphery of diffraction gratings. Although the diffraction grating has been worked, tool marks are clearly visible on the optical surface. The surface roughnesses at the center and at the periphery of the optical surface are 4.9 and 14.9 nm, respectively.

Figures 13 show images of a diamond cutting tool after (a) rough machining and (b) final machining. The dashed lines indicate the tool shape prior to machining. Edge rounding of the cutting tool disappeared after rough machining (Fig. 13(a)). Moreover, there were many weld deposits on the surface of the cutting tool and they were not removed by rough machining. On the other hand, the point of the cutting tool is chipped after final machining (Fig. 13(b)). Furthermore, there are also weld deposits on the cutting tool surface. Thus, although the roughness at the center of the surface is satisfactory for optical devices, the roughness at the outer periphery of the surface is too large because of tool chipping.

In the future, we intend to evaluate the machinabilities of actual length for fabricating a molding die for glass lenses. We will also determine appropriate cutting conditions for each material. Moreover, we intend to search for new Ni–Nb-based amorphous alloys that have better machinabilities.

Fig. 12 (a) Photograph of a molding die for glass lenses and (b), (c) SEM images of the optical surface

Fig. 13 SEM images of diamond cutting tools after (a) rough machining (rounding of the cutting tool) and (b) final machining (point of the cutting tool)

4. Conclusions

We evaluated and compared the characteristics of synthesized and target Ni–Nb–Zr amorphous alloys to determine their suitability as molding die materials for high-precision optical glass lenses. The two samples exhibited no noticeable differences in their linear expansion coefficients, thermal stabilities of their amorphous states and oxidation resistances. On the other hand, they had different mechanical properties and machinabilities due to their different microstructure configurations. The target $\text{Ni}_{35}\text{Nb}_{40}\text{Zr}_{25}$ sample exhibited excellent mechanical properties, including a high fracture stress and a high hardness, a similar linear expansion coefficient to that of BK7, an excellent thermal stability of amorphous state, an acceptable oxidation resistance, and high machinability. Finally, we fabricated an optical glass lens molding die with diffraction gratings made from amorphous $\text{Ni}_{35}\text{Nb}_{40}\text{Zr}_{25}$ alloy. However, we need to clarify the following points:

1. The effect of the sputtering conditions and microstructures on the machinability of the amorphous alloys;
2. Determine suitable cutting conditions for each material;
3. Improve the machinability of Ni–Nb-based amorphous alloys by varying the third element if necessary.

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