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DEVELOPMENT OF MIRROR-POLISHING PROCESS TECHNOLOGY FOR DIFFICULT-TO-POLISH MATERIALS

Mirror-polishing processes have recently been required to add high quality, high-precision precision and multi-functionality to industrial products. However, this requires skilled manual work, making it difficult to improve productivity. Automatic polishing of these products is also very difficult. Furthermore, mirror-polishing process of stainless steel, titanium and glass, which are often used in high-value-added medical products, is also difficult. Therefore, in this study, a technology for high-precision, automatic mirror polishing of three difficult-to-polish materials - stainless steel, titanium and glass - was developed and evaluated. First, the difficult-to-polish properties of different materials were considered, and then tools were developed to improve these properties and enable suitable mirror-polishing processes. Next, a polishing slurry was developed to improve the surface roughness ($Rz = 0.1$ μm or less). Finally, the optimum polishing conditions for the proposed mirror-polishing process were determined by design of experiment and then evaluated. It was concluded that; (1) In order to perform mirror-polishing processes of three types of workpieces, stainless steel (SUS304), titanium (pure titanium) and glass (quartz glass), new polishing tool and slurries corresponding to each material were developed and the optimum polishing processing conditions were clarified, (2) Using (1), mirror-polishing processes of each material with a surface roughness of $Rz = 0.1$ μm has been achieved.

1. INTRODUCTION

Mirror machining has become a very important process for the high quality, high integrity and high performance of industrial products. It has been applied not only in the manufacture of various workpiece materials and industrial products, but also in the development of new machining principles and technological improvements [1]. Especially in medical products, mirror processing is of great importance to improve the functionality of the product [2]. However, this work is still mainly carried out by skilled workers who spend long hours poling [3], which reduces productivity and delays development. In the previous researches, several polishing systems and technologies have been developed [4-6] (see below in Chapter 2).

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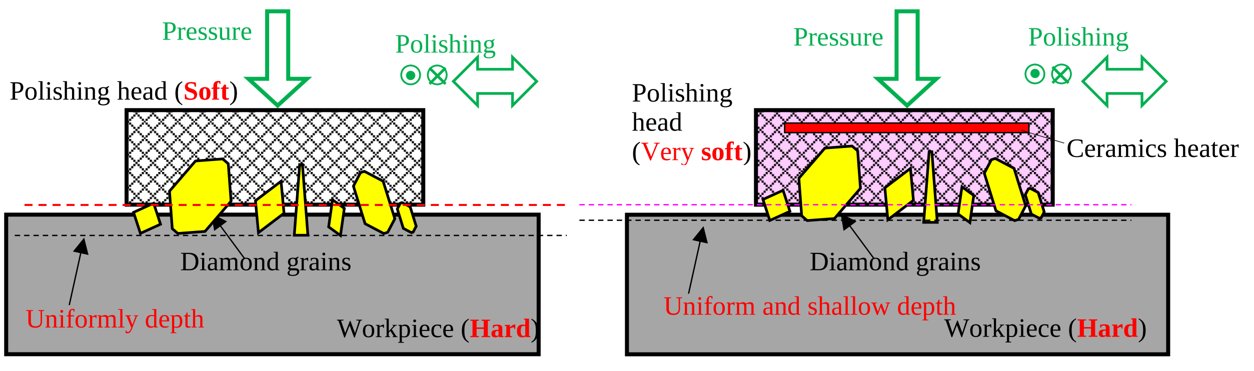
In this research, three workpieces, stainless steel (SUS304), titanium (pure titanium) and glass (quartz glass), which are considered difficult-to-polish materials, were targeted. First, the polishing characteristics of each workpiece were considered and improvement measures were investigated, then the optimal polishing conditions were identified using the design of experiment method, and finally the degree of mirror polishing was evaluated (criteria; maximum height Rz = 0.1 μm or less). Research on increasing the speed of the developed mirror polishing technique will be carried out in a future paper. And this is a technical paper.

2. PRINCIPLES OF POLISHING AND PROBLEMS FOR MIRROR-POLISHING PROCESS OF DIFFICULT-TO-POLISH MATERIALS

2. 1. PRINCIPLE OF THIS POLISHING PROCESS AND THE PROCESSING METHODS DEVELOPED TO DATE USING THEM

Figure 1 shows the basic principle of the polishing process. In Fig. 1a, a soft polishing terminal is pressed against a hard workpiece, and diamond grains in the slurry are interposed between them. For example, by using a soft material such as polypropylene (PP) for the polishing terminal, a hard material such as cemented carbide V10 for the workpiece, and diamond grains as the abrasive grains, the diamond grains can easily penetrate the soft polishing terminal side. This reduces the difference in the amount of push-in to the workpiece side due to the dispersion of the size and shape of the diamond grain, and at the same time makes the amount of push-in to the workpiece uniform. Then, the polishing terminal moves horizontally while holding the pressed the diamond grains securely, and it becomes possible for the diamond grains to scratch the workpiece with a uniform depth. If the difference in the depths machined by the diamond grains is less than 0.1 μm, a mirror surface can be obtained as a result.

In previous work with this basic principle by the author, a polishing tool terminal consisting of several polishing units was developed, and a system for automatic polishing of large parts was developed using this terminal.



(a) Basic principle (b) Polishing principle for improvement of the surface roughness

Fig. 1. Schematic view of polishing principle (When the soft tool presses on the hard workpiece, differences between depths of several diamond grains in the workpiece become uniformly)

A high-speed automatic polishing technique for small parts with a few millimetre-size has been developed using a small linear motor drive [4–6], and a mirror-like polishing technique for soft materials such as plastics has been developed by controlling the temperature of the polishing tool terminals. In addition, technology has been developed for the mirror-finishing of micro-parts with complex shapes. Particularly, in a previous study, it was reported that by reducing the hardness ratio of the workpiece and the tool terminal to less than 5:1, the difference between the maximum and the minimum push of the diamond grain into the workpiece can be reduced to 0.4 μm, thus enabling mirror-like surface polishing. Then, as shown in Fig. 1b, by embedding a ceramic heater in the polishing terminal and heating and softening the polishing terminal, the amount of diamond abrasive pressed into the workpiece becomes shallow, and the difference in the amount of diamond abrasive pressed into the workpiece becomes small accordingly. The difference in the amount of diamond grain pressed into the workpiece is also smaller. This has been used in previous research to polish acrylic workpieces to a mirror-like polishing. As other remarks, it was important for the system to have a stable drive during polishing and to reproduce a uniform polishing pressure. In addition, the particle size and weight ratio of the diamond grains in the slurry are also important for applying scratches of uniform depth to the workpiece. In addition, previous studies have proposed an additive to the slurry (L-ascorbic acid) to prevent oxidation during the polishing process of workpiece titanium and the removal and cleaning of oxides formed during the polishing process of workpiece alumina with the non-woven fabric tool, respectively.

The developed method has carried out mirror polishing of various materials, but finally, stainless steel, titanium and glass are difficult to mirror polish due to their high ductility, high oxidation and high brittleness, respectively. Moreover, mirror polishing of these three materials is required in the production of high-quality, high-precision and high-grade medical devices. Therefore, based on the above principles and experience, this research is developed a high-precision mirror-like polishing technology specifically for improving the surface roughness of medical device materials. Using the polish terminal of a polypropylene head, which has been used in the past, mirror-like polishing of stainless steel (SUS304), titanium (pure titanium) and glass (quartz glass) is carried out to clarify the new problems. These three materials are difficult-to-polishing, and even if conventional methods were applied as they are, the polishing process would be difficult (see section 2.2). Therefore, the tool, slurry and processing method are improved to find the optimum polishing conditions for each workpiece, and the results were evaluated in experiments.

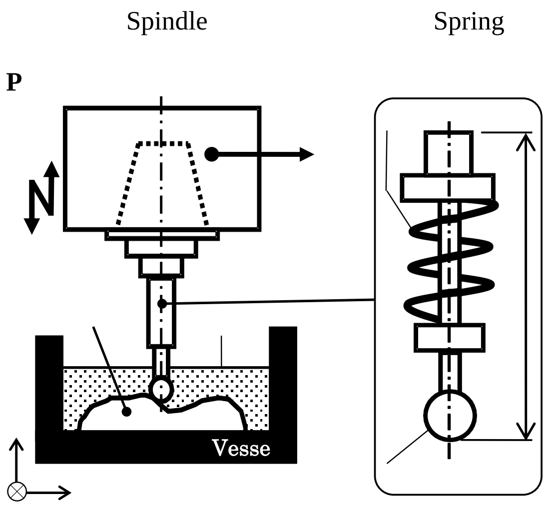
2. 2. PROBLEMS IN MIRROR-LIKE POLISHING OF STAINLESS STEEL, TITANIUM AND GLASS AND THEIR COUNTERMEASURES

First, mirror polishing was carried out on three types of workpieces: stainless steel (SUS304), titanium (pure titanium) and glass (quartz glass), using the optimum machining conditions for cemented carbides revealed in the author's previous research. Table 1 shows the polishing tools used, the machining conditions and the slurry specifications, respectively.

Figure 2 shows the results of laser microscopic observation (photograph) of the machined surfaces of three different workpieces after mirror-like polishing under the machining conditions shown in Table 1. On stainless steel (SUS304), a black area of visible size was observed at the end of the polishing process with a slurry containing diamond grains of mesh size #2500, as shown in the top diagram in Fig. 2a.

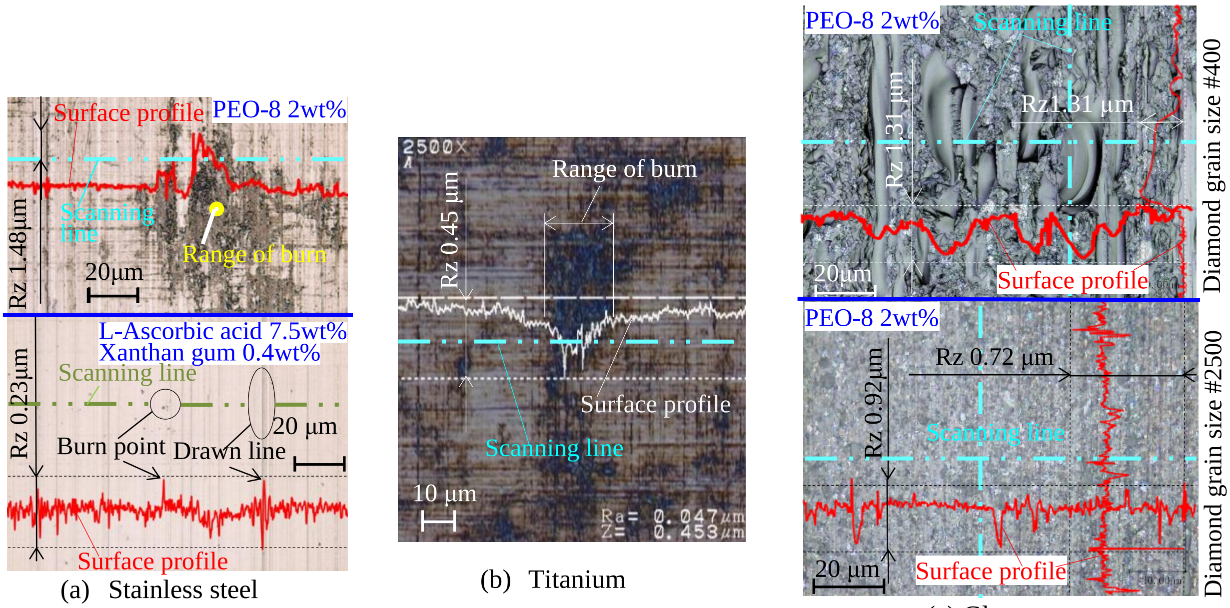
Table 1. Polishing tool, polishing process condition and slurry specification. The cleared conditions for the mirror-like polishing in the previous research. These conditions were used for investigating the problems and the countermeasures of the mirror-like polishing regarding stainless steel, titanium and glass

Polishing tool & polishing system
This spindle is moved by NC control.



Polishing process condition	
Polishing pressure	60 MPa
Polishing speed	3000 mm/min
Polishing pitch	0.1 mm
Polishing area	10 mm × 10 mm
Slurry specification	
Base fluid	Tap water
Ratio of PEO-8	2 wt.%
Ratio of diamond	2 wt.%
Diamond grain mesh size	#400-500, 1200, 2500

Workpiece			
Material	SUS304	Pure titanium	Quartz glass
Surface roughness	Rz1.52μm	Rz1.42μm	Rz 0.09 μm



(a) Stainless steel (b) Titanium (c) Glass

Fig. 2. Photographs on each workpiece with a few faults after polishing process for three medical product materials; stainless steel, titanium and glass. These materials need the several countermeasures to remove the faults

This is thought to be due to the low thermal conductivity of the nickel contained in stainless steel, which causes the material to be thermally affected and oxidised during processing. Similar oxides were also observed during the polishing process of aluminium and titanium in previous studies. As a counter-measure, L-ascorbic acid (7.5 wt%) and xanthan gum (0.4 wt%) were added to the slurry, as shown in the diagram below in Fig. 2a. This suppressed the oxidation phenomenon and the visual black spots were no longer observed, however, small black spots were observed again by laser microscopy, which affected the surface roughness. In addition, the draw lines caused by diamond grains still remain and are a detriment to the improvement of surface roughness. When a slurry containing diamond grains of mesh size #400-500 or #1200 was used, the diamond grains that fell off the polishing tool rolled over the workpiece and caused deep localized scratches. It is thought that this is because the PP polishing head did not have enough force to grip the diamond grains against the large ductility of the stainless steel. In addition, while the polyethylene oxide (hereinafter referred to as PEO) used earlier had the effect of uniformly distributing and retaining the diamond grains, the L-ascorbic acid used as a substitute for PEO had less of an effect, so the surface roughness was thought to be worse than when PEO was used. As a countermeasure, a non-woven fabric (split fiver) made of polypropylene (hereafter PP) and polyethylene terephthalate (hereafter PET) was attached to the polishing tool to strengthen the gripping force, as shown in Fig. 3. In addition, it was decided to reduce the polishing pressure and feed speed, and to use diamond grains to remove the draw lines. This reduction in poling pressure and feed rate simultaneously reduces the oxidation phenomenon caused by cutting heat. In addition, the non-woven fabric used can efficiently remove chips from the polishing process. In order to do this efficiently, the polishing tool is rotated. A slurry containing 2wt% PEO_{-8} was used to ensure uniform distribution and retention of the diamond grains (L-ascorbic acid and xanthan gum were not used). Furthermore, the concentration of diamond grains in the slurry was reduced to ensure that the diamond grains were securely grasped by the polishing tool. Preliminary experiments (Polishing pressure 10 MPa, Polishing speed 500 m/min, Polishing tool with non-woven fabric (see Fig. 3), Polishing tool rotation speed 500 min^{-1} , ratio of diamond in slurry 1.0 wt%) showed that the black dots and drag lines observed earlier could be removed.

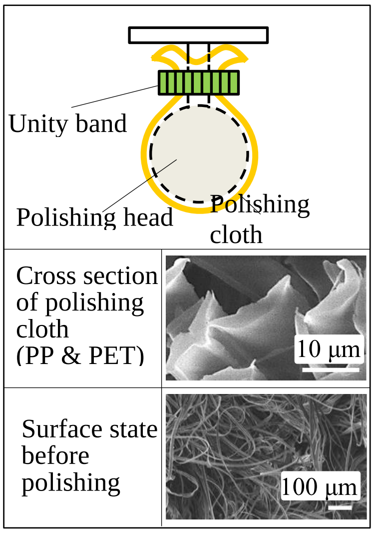


Fig. 3. The improved polishing tool with the nonwoven fabric for mirror-like polishing

Table 2. Each problem and countermeasure; new each sully specification, tool, condition and matters that require attention for mirror-like polishing of the medical product materials; stainless steel, titanium and glass

Workpiece	Stainless steel (SUS304)		Titanium (Pure titanium)	Glass (Quartz)
Problems for mirror-like surface regarding medical product materials	Tenacity and Ductility		Low thermal conductivity	High hardness
	Low thermal conductivity		High oxidation nature	High stiffness
	High oxidation nature			Low brittleness
Improvement for sully and polishing tool		Using: Nonwoven fabric		
		Using PEO-8 and Polishing cloth		Using PEO-8 and Cerium oxide
		Lower diamond ratio		
			Smaller grain size	
Improvement for polishing process condition		Rotation of the polishing tool		No rotation
		Lower polishing pressure		
		Lower polishing speed		

In addition, the polishing pitch was set so that the overlapping contact area was 30% for each polishing pressure. In the next section, the optimum polishing processing conditions are clarified using the design of experiments method.

As shown in Fig. 2b, it is well known that titanium (pure titanium) is a material that is difficult to machine to a mirror finish, as it tends to “burn” during the grinding process. The author's previous study revealed that the black areas were oxidized, and by adding L-ascorbic acid and xanthan gum to the slurry, a surface roughness of Rz = 0.1 μm in the policing direction was already achieved. However, the surface roughness in the direction perpendicular to the policing direction was Rz = 0.147 μm, because the drawn line caused by the policing tool could not be removed. Therefore, in the next chapter, the surface roughness in the polishing direction was set to Rz = 0.1 μm, using the previous study, and then, as in the case of stainless steel, a polishing tool with a non-woven fabric (see Fig. 3) was used to examine the removal of the draw lines. A slurry containing 2 wt.% of PEO_{-8} was also used (L-ascorbic acid and xanthan gum were not used).

In the case of the glass (quartz glass) shown in Fig. 2c, the surface roughness of the glass before machining was Rz = 0.09 μm, and it was already in a mirror-like state. However, I decided to clarify the problem by experiment, assuming that the workpiece that had been pre-processed, such as by grinding, would be polished to a mirror-like surface. When polishing was carried out using a slurry containing diamond grains with a mesh size of #400 (without the addition of cerium oxide), as shown in the upper figure of Fig. 2c, deterioration of the surface roughness was observed as the process progressed. This is due to the fact that the diamond grains that fell off from the polishing tool rolled over the workpiece and caused deep scratches locally, and also due to the fact that glass is a brittle material and cracks were caused by the machining force. In addition, the surface roughness was not improved by brittle fracture even with the slurry containing diamond grains of mesh size #2500 (without cerium oxide addition), as shown in the diagram below in Fig. 2c. As a countermeasure, cerium oxide will be added to the slurry and the glass was polished while moderately softening it. It is also decided to use a design of experiments to find the optimum polishing process conditions that would improve the surface roughness without causing cracking.

Based on the above considerations, new tools, slurries and processing methods for mirror polishing of three difficult-to-polish materials are developed, as shown in Table 2. In accordance with this policy, the optimum polishing process conditions for each workpiece are explored and evaluated in the following chapters.

3. PROPOSAL AND EVALUATION OF MIRROR-LIKE POLISHING PROCESS FOR STAINLESS STEEL, TITANIUM AND GLASS

3. 1. DETERMINATION OF SLURRIES AND OPTIMUM POLISHING CONDITION FOR MIRROR-LIKE POLISHING OF STAINLESS STEEL AND ITS EVALUATION

For the stainless steel SUS304, new tools, slurries and processing methods for mirror polishing are developed using the design of experiment method, the optimum polishing process conditions are determined, and finally their effectiveness is evaluated.

Table 3 shows the control factors and their levels for the design of experiments. Referring to the discussion in the previous chapter, the polishing pressure and polishing speed were set as control factors in consideration of preventing diamond grains from falling off, and the number of nonwoven fabrics and the polishing tool rotation speed were set as control factors for improving the surface roughness. Table 4 shows the remaining polishing conditions, slurry specifications and workpiece specifications. These conditions are the same for all the experiments in this section. Based on the results of a previous study, the polishing pitch was set to a value dependent on the polishing pressure (Table 4) so that the indentations on the polished tools overlap. For this reason, the total polishing time is longer under conditions of slower polishing speed and lower polishing pressure. The surface roughness of the workpiece before mirror polishing is also shown in Table 4, Table 5 shows the L9 orthogonal table of the design of experiments and the experimental results respectively.

The experimental results include the final surface roughness Rz for each diamond grain size (mesh size) used in the slurry and the total polishing time. The total polishing time does not include the set-up time, slurry change and the associated ultrasonic cleaning of the workpiece.

Table 3. Control factors and these levels for improvement of polishing condition and slurry regarding SUS304

Control factors	Levels		
	Level 1	Level 2	Level 3
Polishing pressure MPa	5	10	20
Number of cloth sheets	1	2	3
Polishing speed mm/min	500	1000	1500
Rotation number min^{-1}	0	500	1000

Table 4. Residual polishing condition and slurry specification regarding SUS304

Polishing process condition	
Polishing pitch	0.01(At 5 MPa), 0.02 (At 10 MPa) and 0.03 (At 20 MPa)
Polishing area	10 mm×10 mm
Slurry specification	
Base fluid	Tap water
Ratio of PEO-8	2 wt.%
Ratio of diamond	1 wt.%
Diamond grain mesh size	#400-500, 1200, 2500
Workpiece	
Material	SUS304
Surface roughness	Rz 1.6 μm

Table 5. L9 Orthogonal array table and experimental results for searching optimum mirror-like polishing condition regarding SUS304

	Control factors				Polishing accuracy (Final properties)			
					Surface roughness Rz μm			Total polishing time min
	Polishing pressure MPa	Number of cloth sheet numbers	Polishing speed mm/min	Rotation number min^{-1}	Diamond grain mesh size			
					400/500	1200	2500	
L1	5	1	500	0	0.63	0.27	0.12	360
L2	5	2	1000	500	0.26	0.16	0.06	270
L3	5	3	1500	1000	0.20	0.11	0.09	810
L4	10	1	1000	1000	0.24	0.13	0.07	810
L5	10	2	1500	0	0.71	0.18	0.11	1620
L6	10	3	500	500	0.27	0.14	0.06	540
L7	20	1	1500	500	0.39	0.28	0.09	180
L8	20	2	500	1000	0.22	0.10	0.08	450
L9	20	3	1000	0	0.75	0.23	0.11	270

The results show that the proposed improvement can reduce the surface roughness Rz of stainless steel SUS304 to less than 0.1 μm. It was also possible to remove the black dots and drawn lines observed in Fig. 2a in Chapter 2. It was also possible to confirm that the surface roughness possible to confirm that the surface roughness was similar in the two orthogonal directions. Table 6 shows the optimum conditions for mirror-like polishing of stainless steel SUS304, which were determined by the additivity of the orthogonal table, based on the results of the design of experiments shown in Table 5. Figure 4 shows the relationship between the polishing time and the surface roughness of a stainless steel with the optimum processing conditions in Table 6. Using a slurry with a diamond grain size (mesh size) of #2500, a mirror-like polishing process with a surface roughness of Rz = 0.05 (Standard deviation σ_3 0.003) to 0.06 μm (Standard deviation σ_3 0.006) was achieved. Figure 5 shows a laser micrograph of the surface of the stainless steel after mirror-like polishing, showing that the surface roughness Rz = 0.05 to 0.06 μm was achieved without the deep scratches caused by rolling diamond grains observed in Chapter 2, or the black patterns caused by oxidation.

Table 6. Optimum mirror-like polishing condition and slurry for SUS304

Polishing process condition		Slurry specification	
Polishing pressure	10 MPa	Base fluid	Tap water
Polishing speed	1000 mm/min	Ratio of PEO-8	2 wt.%
Polishing pitch	0.02 mm	Ratio of diamond	1 wt.%
Polishing area	10×10 mm	Diamond grain mesh size	#400–500, 1200, 2500
Numbers of the cloth	3 sheets	Workpiece	
Rotation number	500 min^{-1}	Material	SUS304
		Surface roughness	Rz 1.52 μm

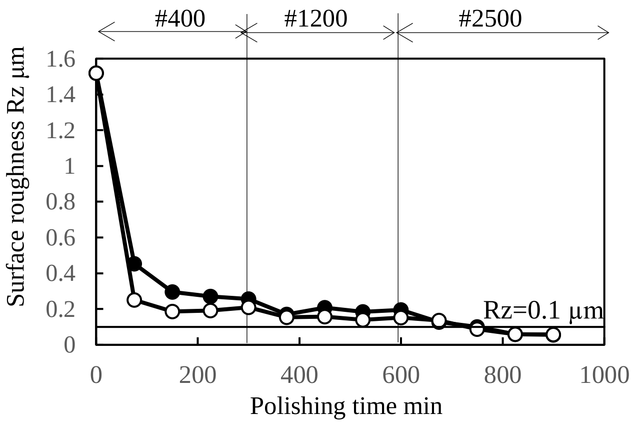


Fig. 4. Relationship between the polishing time and the surface roughness on the workpiece in the polishing process with optimum condition for SUS304. Final surface roughness was Rz 0.05～0.06 μm

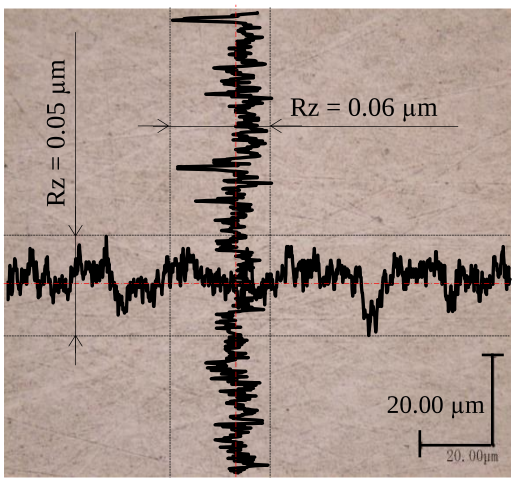


Fig. 5. Photograph on the workpiece after mirror-like polishing using the optimum condition regarding SUS304. Both burn point and drawn line were nothing

The polishing process using the terminal with non-woven fabric proposed in this research is considered. Table 7 shows a comparison of the processing characteristics of grinding, polishing and buffing. The proposed polishing process is positioned between grinding and buffing, based on the rigidity (Young's modulus) of the tools used. As shown in Table 1, when the polish pressure is high with a polypropylene head, it is possible to achieve the same results as when grinding with a resin bonded wheel. As shown in Fig. 3, the polishing process becomes closer to buffing as the number of non-woven fabrics attached to the terminals increases. Therefore, the number of nonwovens is an important control factor for the processing accuracy (dimensional accuracy, shape accuracy, and surface roughness) because the position of this polishing process varies between grinding and buffing depending on the number of nonwovens.

Table 7. Machining properties for grinding, our polishing and buffing. Our polishing was posited between the grinding and the buffing. Position of our polishing was controlled by the number of the polishing cloth

Kinds of machining		Grinding		Our polishing for minute convex	Buffing
		Metal or Vitrified bond wheels	Resin bond wheel		
Young' modulus of tool		Very large	Large	Small	Very small
Grip force of a grain		Very large	Large	Small	Very small
Productivity		Usually	Small	Very small	More very small
Accuracy	Dimension	Good	Excellent	This research!	Fault
	Shape	Good	Excellent	Probably between	Fault
	Surface roughness	Good	Fine (Rz 2 μm)	The grinding and the buffing	Mirror-like surface (Rz 0.1 μm under)

3. 2. DETERMINATION OF OPTIMUM POLISHING CONDITION FOR MIRROR-LIKE POLISHING OF TITANIUM AND ITS EVALUATION

First, for titanium (pure titanium, JIS type2), using the polishing conditions of the previous study, the diamond grain size in the slurry was changed to #400, #1200, #2500, #5000, and #14000 to confirm the reproducibility of the previous study. Figure 6 shows a photograph of the surface after the polishing process. The surface roughness in the polishing direction was Rz = 0.13 μm, and the surface roughness in the perpendicular direction was Rz = 0.15 μm. Furthermore, when the workpiece shown in Fig. 6 was continuously polished (for 15 minutes) with a diamond grain diameter of #5000 in the slurry, the surface roughness deteriorated, as shown in Fig. 7. This is thought to be the effect of the surface of the polishing tool coming into direct contact with the workpiece, whereas when the diamond grain was large, the surface of the polishing tool was not in contact with the workpiece at Fig. 3. In this case, L-ascorbic acid (7.5 wt%) and xanthan gum (0.4wt%) were used in the slurry instead of PEO_{-8}. Then, using a polishing tool with a non- woven fabric (see Fig. 3) as in the case of stainless steel in the previous section, we investigated the polishing process to remove the drawn lines shown in Fig. 6 and to reduce the surface roughness to less than Rz = 0.1 μm in both perpendicular directions. The optimum polishing process conditions for removing drawn lines were also clarified using a design of experiments method.

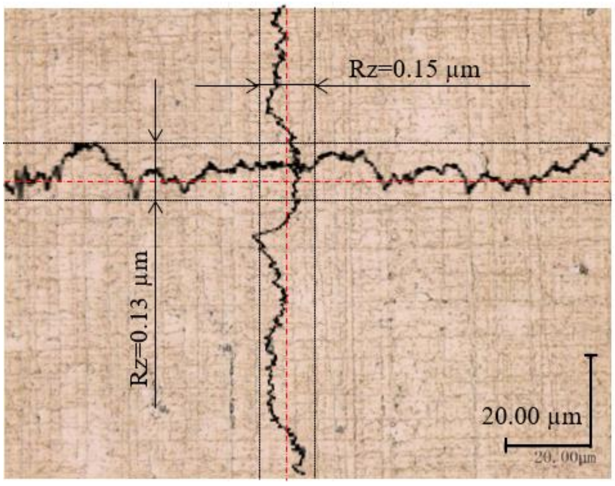


Fig. 6. Photograph on the workpiece (Titanium) after mirror-like polishing using the previous research. Used diamond grains are #400, #1200, #2500, #5000, #14000. Surface roughness on the polishing direction is Rz = 0.13 µm, however, surface roughness on the right angle direction is Rz = 0.147 µm because of the influence of the several polishing draw lines

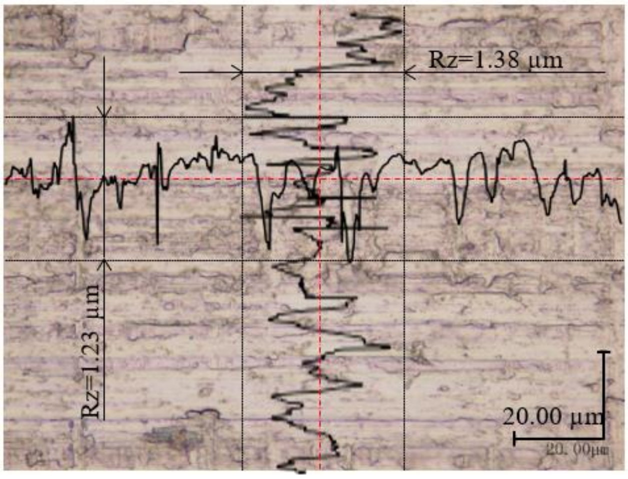


Fig. 7. Photograph on the workpiece (Titanium) after mirror-like polishing using the previous research. Used diamond grain is #50000 after Fig. 6. Surface roughness on the workpiece after mirror-like polishing using the diamond grains #50000. Because the polishing head has contact to the workpiece, surface shape of the polishing head has influence for surface roughness on the workpiece

The control factors and their levels for the design of experiments are given in Table 8, and the remaining polishing conditions are given in Table 9. Again, the polishing pitch was set to a value dependent on the polish pressure (set so that the overlapping contact area was 30%). The workpiece was made of titanium (pure titanium, JIS type 2), and the workpiece before machining was the final workpiece from a previous study (see Fig. 6). Only #14000 diamond grain size in the slurry was used. Table 10 shows the L9 orthogonal table and the experimental results. The experimental results include the surface roughness Rz and the total polishing time in the direction of and perpendicular to the polishing direction. The total polishing time does not include the set-up time, the slurry change and the associated ultrasonic cleaning of the workpiece.

Table 8. Control factors and these levels for improvement of polishing condition and slurry regarding Titanium

Control factors	Levels		
	Level 1	Level 2	Level 3
Polishing pressure MPa	5	10	30
Number of cloth sheets	1	2	3
Polishing speed mm/min	500	1000	1500
Rotation number min^{-1}	500	1000	1500

Table 9. Residual polishing condition and slurry specification regarding Titanium

Polishing process condition	
Polishing pitch	0.01(At 5 MPa), 0.02 (At 10 MPa) and 0.05 (At 30 MPa)
Polishing area	10 mm×10 mm
Slurry specification	
Base fluid	Tap water
Ratio of PEO-8	2 wt.%
Ratio of diamond	1 wt.%
Diamond grain mesh size	#14000
Workpiece	
Material	Pure titanium
Surface roughness	Rz 0.130, 147 µm

Table 10. Orthogonal array table and experimental results for searching optimum mirror-like polishing condition regarding Titanium

	Control factors				Polishing accuracy (Final properties)		
					Surface roughness Rz μm		Total polishing time min
	Polishing pressure MPa	Number of cloth sheet numbers	Polishing speed mm/min	Rotation number min^{-1}	Polishing direction	Right angle direction	
L1	5	1	500	500	0.16	0.14	540
L2	5	2	1000	1000	0.18	0.18	270
L3	5	3	1500	1500	0.09	0.03	180
L4	10	1	1000	1500	0.11	0.14	150
L5	10	2	1500	500	0.38	0.27	90
L6	10	3	500	1000	0.05	0.04	270
L7	30	1	1500	1000	0.37	0.30	48
L8	30	2	500	1500	0.10	0.07	120
L9	30	3	1000	500	0.27	0.18	60

The experimental results show that the proposed improvement reduces the surface roughness Rz of titanium (pure titanium, JIS type 2) to less than 0.1 μm. It was also confirmed that the surface roughness was similar in two orthogonal directions. It was also possible to remove the black dots and drawn lines observed in Fig. 2b in Chapter 2. As for the fact that the black area observed in Chapter 2 did not occur even without the addition of L-ascorbic acid and xanthan gum to the slurry (PEO-8 was used), it is thought that this is because the generated oxide was wiped off by the non-woven fabric of the polishing tool.

Table 11 shows the optimum conditions for mirror polishing of titanium (pure titanium, JIS type 2) to remove drawn lines, calculated by the additivity of the orthogonal table based on the results of the design of experiments shown in Table 10. Figure 8 shows the relationship between the polishing time and the surface roughness when the optimum conditions in Table 12 were used to polish the titanium surface to remove drawn lines. For reference, the machi-ning conditions of the previous study were used, and the results of pre-processing with varying diamond grain diameters of #400, #1200, #2500, #5000 and #14000 in the slurry are shown in the grey range. The surface roughness Rz = 0.03 to 0.04 μm in two orthogonal directions was achieved by mirror polishing under the optimum machining conditions shown in Table 11. Figure 9 shows a laser micrograph of the titanium surface after the mirror-like polishing process, showing that the surface roughness in two orthogonal directions, Rz = 0.03 (Standard deviation σ_3 0.005) to 0.04 μm (Standard deviation σ_3 0.003), was achieved without the black pattern caused by oxidation observed.

Table 11. Optimum mirror-like polishing condition and slurry for Titanium

Polishing process condition		Slurry specification	
Polishing pressure	5 MPa	Base fluid	Tap water
Polishing speed	500 mm/min	Ratio of PEO-8	2 wt.%
Polishing pitch	0.01 mm	Ratio of diamond	1 wt.%
Polishing area	10×10 mm	Diamond grain mesh size	# 14000
Numbers of the cloth	3 sheets	Workpiece	
Rotation number	1500 min^{-1}	Material	Pure titanium
		Surface roughness	Rz 0.130, 147 μm

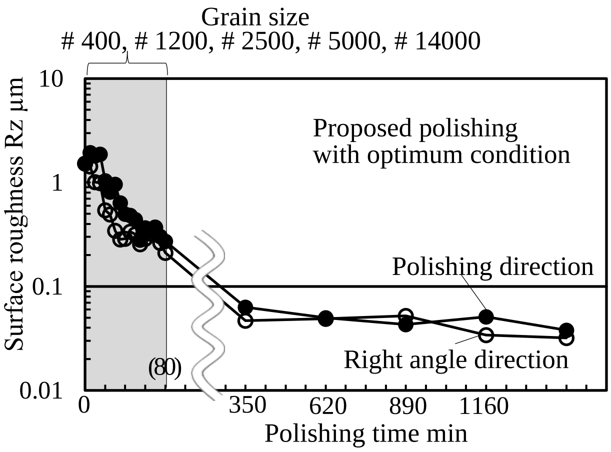


Fig. 8. Relationship between the polishing time and the surface roughness on the workpiece in the polishing process with optimum condition for Titanium. Final surface roughness was Rz 0.033～0.038 µm.

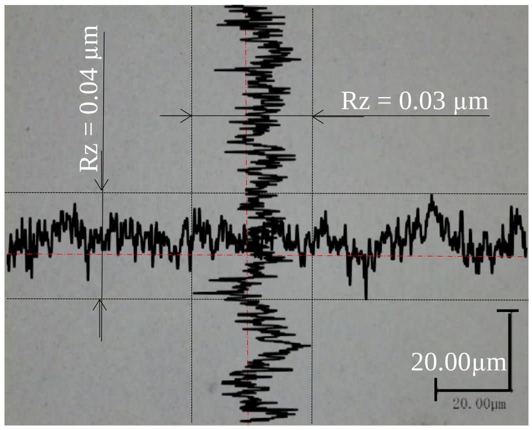


Fig. 9. Photograph on the workpiece after mirror-like polishing using the optimum condition regarding Titanium. Both burn point and drawn line were nothing

3. 3. DETERMINATION OF SLURRIES AND OPTIMUM POLISHING CONDITION FOR MIRROR-LIKE POLISHING OF GLASS AND ITS EVALUATION

Based on the discussion in the previous chapter, a new slurry for mirror-like polishing of quartz glass is developed, and the optimum polishing conditions are determined and evaluated using the design of experiments method. In this section, the possibility of mirror polishing with a slurry containing cesium oxide was investigated by preparing a workpiece made of quartz glass with a surface roughness of Rz of 0.9 µm. As in the previous case of stainless steel and titanium, the surface roughness did not improve when the polishing process was carried out at low polishing pressure and low polishing speed using the polishing tool with non-woven fabric. Therefore, it was decided to carry out the polishing process using the polishing tool (without non-woven fabric) shown in Table 1 at high polishing pressure and high polishing speed. The reason for this is that even though quartz glass is softened by cerium oxide, it still has the characteristics of a hard material, and we thought that the processing conditions used.

Table 12 shows the control factors and their levels for the design of experiment method. The first control factor was a set of polishing pressures and speeds, referring to the mirror-like polishing conditions for cemented carbides in Table 1. The remaining three control factors were the weight ratio of each of the components of the slurry: polymer PEO_{-8}, cerium oxide and diamond grain. Each level value was determined by referring to Table 1 and previous studies. Since there was no reference material for the weight ratio of cerium oxide, preliminary experiments were conducted, and it was confirmed that polishing processing of quartz glass was possible up to a cerium oxide weight ratio of 40wt% in the slurry, and that the speed of improvement in surface roughness increased as this weight ratio increased. However, when the weight ratio of cerium oxide in the slurry exceeded 10wt%, condensation of cerium oxide and uneven dispersion of diamond grains occurred in the slurry, which gradually made stable polishing difficult.

Table 13 shows the remaining polishing process conditions, slurry specifications, and workpiece specifications. Again, the polishing pitch was set to a value dependent on the polishing pressure (set so that the overlapping contact area is 30%). No non-woven fabrics were used, and the polishing terminals were not rotated.

Table 12. Control factors and these levels for improvement of polishing condition and slurry regarding quartz glass

Control factors		Level 1	Level 2	Level 3
Polishing condition	Pressure MPa	30	45	60
	Speed mm/min	2000	2500	3000
Slurry	PEO-8 wt.%	1	2	4
	Cerium oxide wt.%	5	7.5	10
	Ratio of diamond wt.%	1	2	4

Table 13. Residual polishing condition and slurry

Polishing process condition	
Polishing pitch	0.05(At 30 MPa), 0.08 (At 45 MPa) and 0.1 (At 60 MPa)
Polishing area	10 mm ×10 mm
Slurry specification	
Base fluid	Tap water
Diamond grain mesh size	#2500 and #5000
Workpiece	
Material	Quartz glass
Surface roughness	Rz 0.9 µm

Table 14. L9 Orthogonal array table and experimental results for searching optimum mirror-like polishing condition for quartz glass

	Control factors					Polishing accuracy (Final properties)				
	Polishing condition		Slurry			Surface roughness Rz µm				Total polishing time min
						Grain size #2500		Grain size #5000		
	Pressure MPa	Speed mm/min	PEO-8 wt.%	Cerium oxide wt.%	Ratio of diamond wt.%	Polishing direction	Right angle direction	Polishing direction	Right angle direction	
L1	30	2000	1	5	1	0.99	0.86	0.62	0.52	306
L2	30	2000	2	7.5	2	1.03	0.94	0.59	0.48	306
L3	30	2000	4	10	4	0.96	0.93	0.62	0.50	306
L4	45	2500	1	7.5	4	0.91	0.68	0.13	0.09	198
L5	45	2500	2	10	1	0.97	0.82	0.10	0.09	220
L6	45	2500	4	5	2	0.81	0.65	0.55	0.42	198
L7	60	3000	1	10	2	0.50	0.39	0.07	0.05	162
L8	60	3000	2	5	4	0.89	0.79	0.35	0.26	162
L9	60	3000	4	7.5	1	0.73	0.72	0.19	0.20	162

Table 15. Optimum mirror-like polishing condition and slurry regarding quartz glass

Polishing process condition		Slurry specification	
Polishing pressure	60 MPa	Base fluid	Tap water
Polishing speed	3000 mm/min	PEO-8	1 wt.%
Polishing pitch	0.1 mm	Cerium oxide	10 wt.%
Polishing area	10×10 mm	Diamond grain mesh size	#2500 and #5000
Numbers of the cloth	Nothing	Ratio of diamond	1 wt.%
Rotation number	0 min^{-1}	Workpiece	
		Quartz glass with surface roughness Rz 0.9 µm	

Table 14 shows the L9 orthogonal table and the experimental results. The surface roughness (maximum height) Rz in the final feed direction and perpendicular to it, and the total polishing process time were measured. The total polishing time does not include the setup time, slurry change, and associated ultrasonic cleaning of the workpiece. Table 15 shows the optimum polishing conditions and slurry specifications obtained by applying the additivity of the orthogonal table based on the results of the design of experiments shown in Table 14.

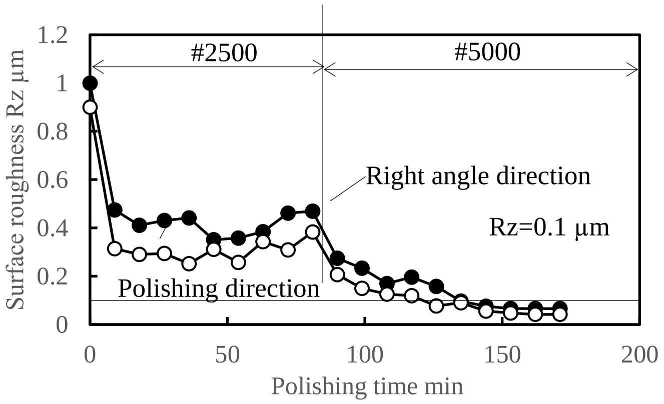


Fig. 10. Relationship between the polishing time and the surface roughness in the polishing process with optimum condition regarding quartz glass

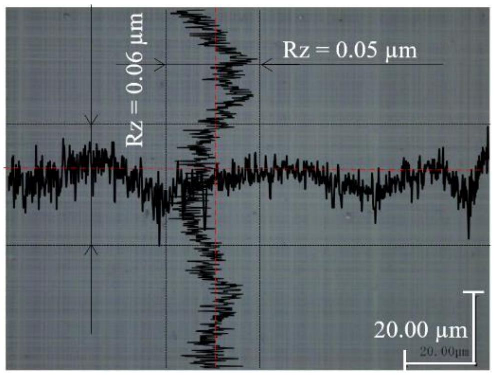


Fig. 11. Photograph on the workpiece after mirror-like polishing using the optimum condition regarding quartz glass

Figure 10 shows the surface roughness when mirror polishing was performed on quartz glass (surface roughness Rz 0.9 μm) using the optimum processing conditions and slurries shown in Table 15. By adding 10 wt.% cesium oxide to the slurry, the deep scratches and brittle fracture caused by the rolling of diamond grains in Fig. 2c were eliminated, and a surface roughness of Rz = 0.1 μm or less could be achieved. Figure 11 shows a laser micrograph of the quartz glass surface after the mirror-like polishing process, which shows that there are no deep scratches or brittle fractures caused by the rolling of diamond grains observed in the previous chapter, and that a clean mirror polishing process with a surface roughness of Rz = 0.05 (Standard deviation σ_3 0.004) to 0.06 μm (Standard deviation σ_3 0.007) was achieved.

As described above, we have developed new slurries and searched for optimal polishing conditions for difficult-to-polish materials such as stainless steel (SUS304), titanium (pure titanium) and glass (quartz glass), and have succeeded in achieving mirror polishing with a surface roughness of less than 0.1 μm for all of these materials.

4. CONCLUSION

The results of this study can be summarized as follows ; (1) In order to perform mirror-like polishing of three types of workpieces, stainless steel (SUS304), titanium (pure titanium) and glass (quartz glass), new slurries corresponding to each material and optimum polishing processing conditions were clarified, (2) Using (1), mirror polishing of stainless steel

(SUS304), titanium (pure titanium) and glass (quartz glass) with a surface roughness of Rz = 0.1 μm has been achieved, (3) The use of a non-woven fabric as a polishing tool has enabled the oxidation of stainless steel and titanium to be prevented and the removal of drawn lines to be achieved, (4) By mixing cerium oxide in the slurry, brittle fracture of the glass caused by the polishing process could be suppressed.

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