



Ultra-precision fabrication of a nickel-phosphorus layer on aluminum substrate by SPDT and MRF

YANG BAI,* ZHIYU ZHANG,  DONGLIN XUE, AND XUEJUN ZHANG

Key Laboratory of Optical System Advanced Manufacturing Technology, Changchun Institute of Optics, Fine Mechanics and Physics, Chinese Academy of Sciences, Changchun 130033, China

*Corresponding author: baiyang5406@sina.com

Received 3 July 2018; revised 7 October 2018; accepted 7 November 2018; posted 9 November 2018 (Doc. ID 336408); published 30 November 2018

Metal mirrors are rarely used in visible or ultraviolet systems due to the ultra-precision fabrication difficulties. In this work, a plane aluminum alloy substrate ($\Phi 100$ mm) surface deposited with a nickel-phosphorus (NiP) layer by the electroless deposition technique is prepared. The NiP layer is processed by single point diamond turning (SPDT) technology to the accuracy of 60 nm in RMS, and the surface roughness reaches 4.157 nm in Ra. A kind of water-based magnetorheological polishing fluid for the ultra-precision of the NiP layer is developed, and magnetorheological finishing (MRF) is applied to the final finishing of the mirror. The developed fluid that contains small size (1.5 μm) carbonyl iron powder (CIP) and 50 nm nano-cerium possesses material removal of 1.8 $\mu\text{m}/\text{min}$, and surface roughness of 1 nm is determined as the optimal fluid formula. The surface residual error is improved from 60 to 10 nm, and the surface roughness decreases from 4.157 to 0.851 nm after MRF in 1.5 h with one polishing cycle with the developed MR polishing fluid. Finally, the surface quality after MRF is tested by SEM and XRD, and the results manifest that the periodical tool mark is wiped out and the surface is not contaminated by MR polishing fluid. The experiment results and theoretical analysis of this work prove that MRF can satisfy the ultra-precision fabrication of NiP film on the metal mirror, and the surface quality can be applied in a visible or even ultraviolet optical system by using suitable MR polishing fluids. © 2018 Optical Society of America

<https://doi.org/10.1364/AO.57.000F62>

1. INTRODUCTION

Ultra-precise metal mirrors are key components of sophisticated scientific instrumentation in astronomy and space applications, covering a wide spectral range [1,2]. Especially for applications in the visible or ultraviolet spectral ranges, low roughness of the optics is required. Metal mirrors are often made of nickel-phosphorus (NiP) plated aluminum alloy Al6061 to reduce light scattering [3,4]. Typically, these layers are approximately 30–60 μm thick. The nano-precision metal mirrors can be obtained by single crystal diamond turning (SPDT). However, the surface of the soft NiP layer will have residual periodical cutting marks after SPD, which will increase the light scattering and decrease the image quality of the system. Therefore, the applications of metal mirrors are naturally limited to the infrared spectral region due to scatter losses for shorter wavelengths, and achieving diffraction limited performance in the visible or ultraviolet spectral ranges demands the application of additional polishing steps [5–8].

NiP layers enable several polishing techniques, e.g., chemical mechanical polishing (CMP) and ion beam figuring for reducing the roughness of the optical surface. However, the process efficiency is not satisfactory. At the same time, magnetorheo-

logical finishing (MRF) has been used to reduce the shape deviation or tool marks remaining on the metal mirrors [9–11]. The application of MRF as a figuring tool for precise metal mirrors is a nontrivial task since the technology was primarily developed for figuring and finishing a variety of other optical materials, such as glasses, the silicon modification layer, and silicon carbide [12–14]. As a consequence of ongoing research and investigation, a variety of other materials, including glass ceramics such as Zerodur, plastics, and even hard polycrystals, were examined to be figured and finished very well with MRF [15,16]. However, the ultra-precision mirror used in visible or ultraviolet spectral ranges by using MRF has rarely been reported. Investigations about the effect of MR polishing fluids' composition on the NiP layer polishing performance, such as the material removal rate, surface roughness, and cleanliness, have not been reported yet, to our knowledge.

In this paper, we determined a kind of MR polishing fluid for NiP layer polishing by carrying a removal function experiment on the NiP deposited layer on Al 6061. By means of investigating the material removal rate and surface roughness, a suitable fluid that possesses relative high removal and good roughness is determined, and the theoretical analysis is

also made according to the material removal mechanism of MRF. The final finishing of the preprocessed NiP layer deposited on a plant Al 6061 mirror by SPDT is fulfilled by MRF with the developed MR polishing fluid. The surface error RMS is improved from 60 to 10 nm, and the surface roughness Ra is improved from 4.157 to 0.851 nm by one polishing cycle in 1.5 h. Surface quality is detected by SEM and XRD, and the results proved that the surface after MRF is smooth and uncontaminated. The results of this work prove that MRF can satisfy the ultra-precision fabrication of the NiP layer on a metal mirror, and the surface quality can be applied in visible or even ultraviolet optical systems by using suitable MR polishing fluid.

2. PROCESSING OF NIP LAYER BY SPDT

The metal mirror material of this work is Al 6061, which has the advantages of easy processing, light weight, high thermal conductivity, and low cost compared to other mirror materials such as silicon carbide or beryllium [4]. The Al 6061 substrate surface is processed by SPDT to surface error of nearly 30 nm in RMS. Then the surface is deposited by the NiP layer that is created by the electroless method. The thickness of the NiP layer is about 10 μm to reduce the bimetallic effect, caused by different coefficients of thermal expansion of Al 6061 and NiP [3,17]. The processing photograph is shown in Fig. 1.

To achieve good surface accuracy and roughness, we selected a small diamond turning tool, high spindle rotation speed, and small cut depth. The specific processing parameters are shown in Table 1.

The surface residual error after one cutting cycle is measured with a Zygo interferometer, and the surface roughness is mea-



Fig. 1. Process of NiP layer by SPDT.

Table 1. Processing Parameters of SPDT

Parameters	Spindle Rotation Speed	Cutting Tool Radius	Cutting Depth
Value	2000 r/min	1.032 mm	2 μm

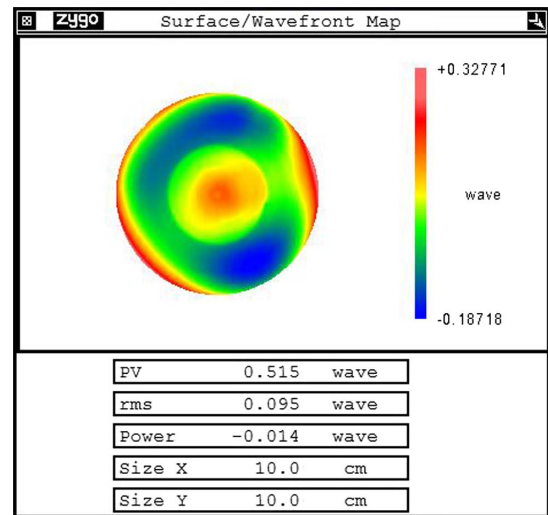


Fig. 2. Surface residual errors after SPDT.

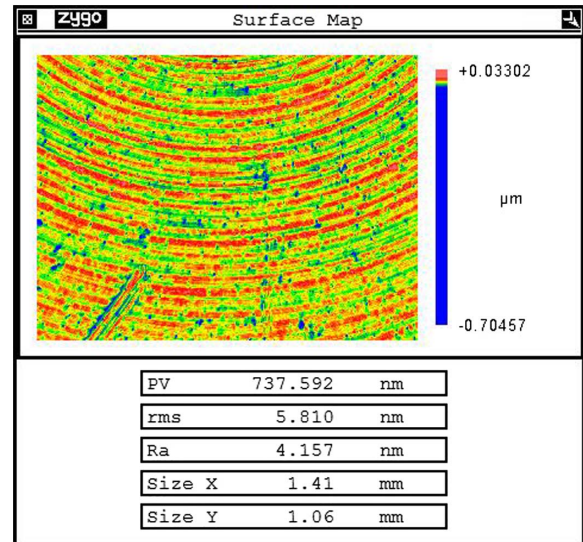


Fig. 3. Surface roughness after SPDT.

sured with the Zygo New View 7200 white-light interferometer. The results are shown in Figs. 2 and 3, respectively.

The results illustrate that the surface residual error is 60 nm in RMS with big low-frequency surface error, and the surface roughness is 4.157 nm with obvious periodical tool marks after being processed with SPDT. The post-polishing process is necessary to improve the surface accuracy and roughness. MRF is the hopeful process technology if the appropriate MR polishing fluid and polishing parameters are used.

3. MR POLISHING FLUID FOR NIP LAYER

At present, many kinds of materials can be polished with aqueous MR polishing fluid such as glasses, silicon, and silicon carbide [13,17–22]. Due to the mechanical properties of

metal, such as soft, flexible, or magnetic conducting, there are few research reports about fluid suitable for a metal or alloy layer. Therefore, to achieve an ultra-precision surface of the NiP layer and make the metal mirror widely used in a visible or ultraviolet optical system, developing an MR polishing fluid suitable for the finishing of the surface of the NiP layer is very significant.

In consideration of the mechanical properties of NiP, we select nano-Zirconia, nano-alumina, and polycrystalline nano-diamond as the abrasives. The diameters of all the abrasives are 50 nm. For iron powder (carbonyl iron), we select 4 μm , which is commonly used in conventional fluid, and 1.5 μm , which is customized for use in this work. Removal function experiments are carried out to investigate the polishing performance of six different types of MR polishing fluids. The powder ingredients of the fluids are listed in Table 2.

The experimental parts in this work are in the same preparation condition. The removal function experiment parameters are listed in Table 3.

The equipment used in the experiment is developed by our research group and the magnetic field structure using a permanent magnet. The experiment photograph is shown in Fig. 4.

The peak removal rate (PRR) of six fluids is shown in Fig. 5.

From the results, it can be seen that the fluids with 4 μm CIP possess higher removal rate than fluid with 1.5 μm carbonyl iron, although the abrasives are the same. This provides that the diameter of carbonyl iron has a great effect on the material removal rate. The highest PRR comes from fluid No. 3, and the lowest is from No. 4. The surface roughness after polishing with six kinds fluids is also investigated. The results are shown in Fig. 6.

As shown in Fig. 6, the roughness results are contrary to the material removal rate. The fluids that use 4 μm CIP produce higher roughness than fluids with 1.5 μm carbonyl iron, although the abrasives are the same. These results can be explained with the previous work by Kordonsiki, who proposed

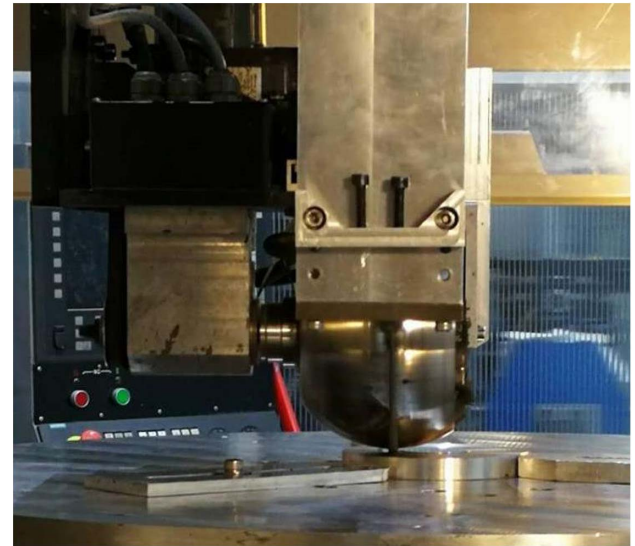


Fig. 4. Experimental diagram and MRF equipment.

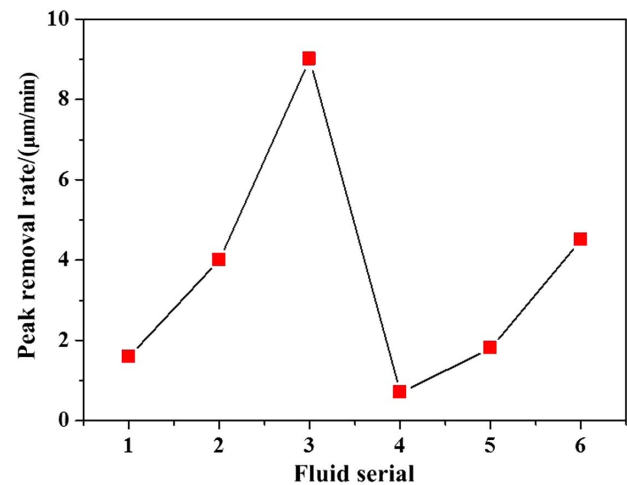


Fig. 5. Peak removal rate of six fluids.

Table 2. Powder Ingredients of Six Fluids

No.	Iron	Iron	Abrasives	Abrasives
	Diameter/ μm	Volume Concentration (%)		Volume Concentration (%)
1	4	37	Nano-zirconia	0.2
2	4	37	Nano-alumina	0.2
3	4	37	Nano-diamond	0.2
4	1.5	37	Nano-Zirconia	0.2
5	1.5	37	Nano-alumina	0.2
6	1.5	37	Nano-diamond	0.2

Table 3. Experiment Parameters

Parameters	Wheel Diameter	Rotation Speed	Dwell Time	Penetration Depth
Value	160 mm	2 r/min	5 s	1.0 mm

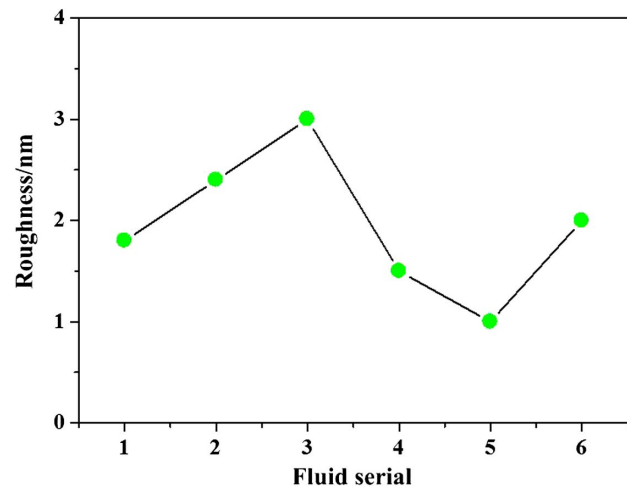


Fig. 6. Surface roughness of six fluids.

that the material removal of MRF stems from the granular collision between iron particles and abrasives [23]. The material removal rate and surface quality strongly depend on the granular force and particle properties. The force that brings about material removal is derived from sheared CIPs' impact with abrasives. The force of CIPs takes the form of

$$G_p = K \cdot \frac{\pi}{4} \cdot \rho_p \cdot d_p^4 \cdot \dot{\gamma}^2 \tag{1}$$

ρ_p is the density of CIP, d_p is the diameter of CIP, and $\dot{\gamma}$ is the shear rate. The dimensionless coefficient K takes into account other flow parameters such as the concentration, mechanical properties of particles, carrier fluid damping properties, and flow geometry. It can be seen from Eq. (1) that the large size of CIPs (d_p) will produce a more powerful processing force than small size CIPs in the polishing process. Based on the Hertzian theory [24], the tensile stress generated over the contact area is given by

$$\sigma_p = \frac{(1 - 2 \cdot \vartheta_M) \cdot G_p^{\frac{1}{3}}}{2 \cdot \pi \cdot r_c^2} \left(\frac{3}{4} r_a \cdot K_E \right)^{-\frac{2}{3}} \tag{2}$$

$$K_E = \left(\frac{1 - \vartheta_M^2}{E_M} + \frac{1 - \vartheta_a^2}{E_a} \right) \tag{3}$$

where r_a is the radius of the abrasive particle, ϑ_M and E_M are the Poisson's ratio and Young's modulus for the material, and ϑ_a and E_a are Poisson's ratio and Young's modulus for the abrasive particle. K_E is the reciprocal of reduced elastic modulus.

It can be seen from Eqs. (2) and (3) that the polished material mechanical property, abrasive mechanical property, and size also influence the material removal rate and surface roughness. Based the removal function experiment results, fluid No. 5, which used small size CIPs and nano-alumina, possesses good roughness and a relatively high peak material removal rate, because of the small processing force and moderate abrasive size and mechanical properties.

Then, we can infer that the ultra-precision mirrors that satisfy the visible image system or even the ultraviolet

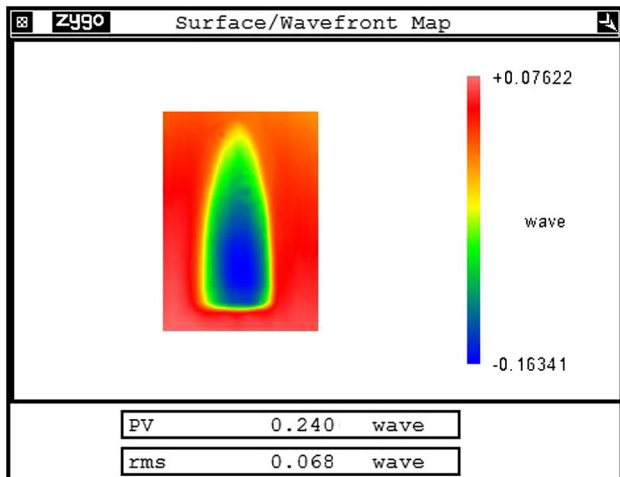


Fig. 7. Removal function of fluid No. 5.

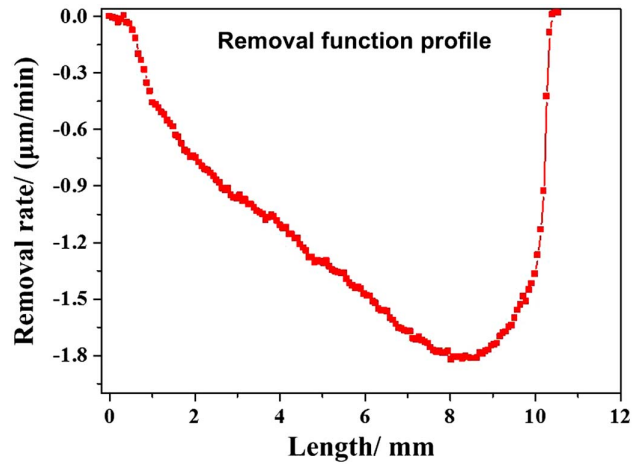


Fig. 8. Removal function profile of fluid No. 5.

system demand can be finished by MRF with suitable fluid ingredients. The removal function results with fluid No. 5 are shown in Figs. 7 and 8. The volume removal rate that is calculated based the pixel resolution of the interferometer is $3.7 \times 10^7 \mu\text{m}^3/\text{min}$. Then, the removal function of fluid No. 5 is applied to the calculation of the dwell time matrix in the subsequent final finishing of the NiP layer.

4. FINAL FINISHING OF NIP LAYER

The final finishing of the NiP layer on the Al 6061 ($\Phi 100$ mm) plane mirror is carried out with the same equipment and parameters as the removal function experiment used, and the raster trajectory is used with 0.5 mm step length. The results of the surface residual error and roughness after one polishing cycle with MRF in 1.5 h are shown in Figs. 9 and 10. As

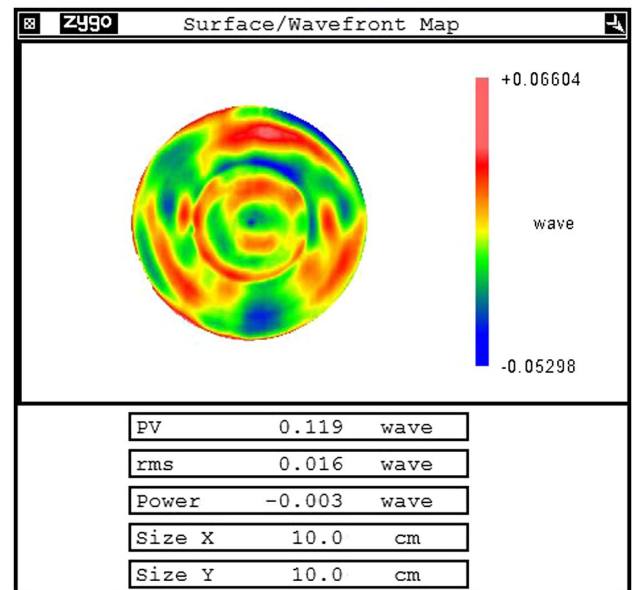


Fig. 9. Surface residual error after MRF.

the result shown in Fig. 9, the surface residual error is improved from 60 to 10 nm after MRF in 1.5 h with one polishing cycle. The convergence rate reaches 77%, which proves the high processing efficiency of MRF and good performance of the developed MR polishing fluid. The roughness results shown in Fig. 9 prove that the periodical tool marks of SPDT are all swiped out and the surface roughness Ra is improved from 4.157 to 0.851 nm in a 1.41 mm × 1.06 mm area scope.

5. SURFACE MORPHOLOGY AND CLEANLINESS TEST

The surface morphology and cleanliness of the NiP layer after MRF are also tested. The testing results are shown Figs. 11 and 12. As shown in Fig. 10, the SEM results manifest that the periodical tool marks are all eliminated without any remaining of scratches of MRF. The XRD result shown in Fig. 12 requires

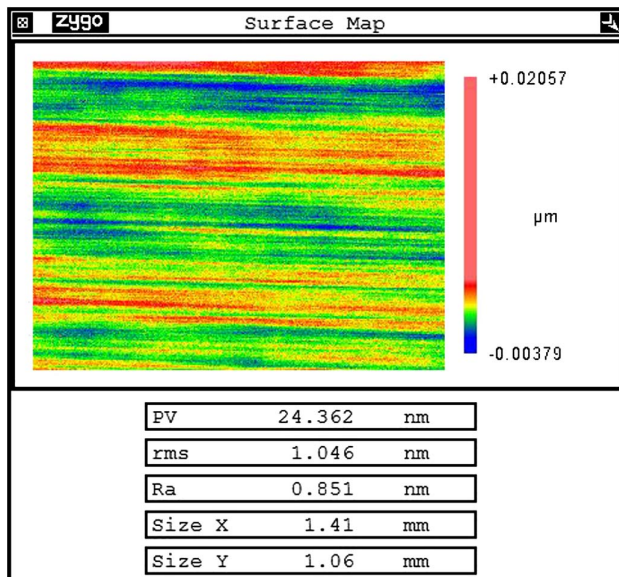


Fig. 10. Surface roughness after MRF.

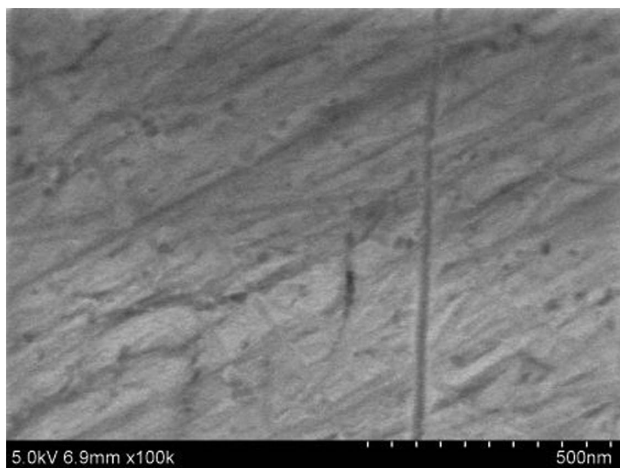


Fig. 11. SEM image of surface after NiP layer after MRF.

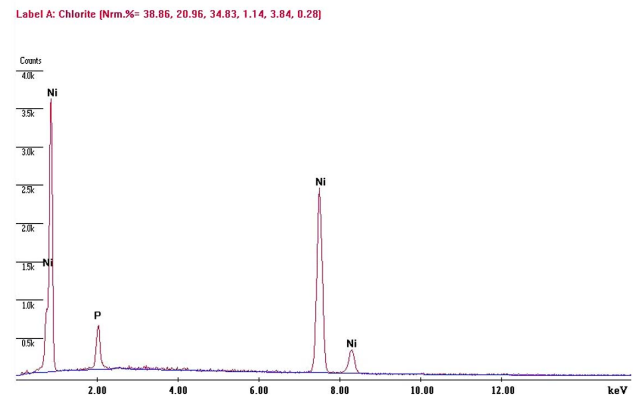


Fig. 12. XRD result of NiP layer surface after MRF.

that the element composition of the NiP surface after MRF only includes nickel and phosphorus. The composition of the MR fluid including carbonyl iron and chemical agents is easily cleaned without contamination.

6. SUMMARY

In this paper, MR polishing fluid that is suitable for the polishing of NiP is obtained by the experimental study of six different fluid compositions. The fluid that possesses a relatively high removal rate and good surface roughness is applied to the final finishing of the NiP layer on Al 6061 after SPDT. The surface residual error is improved from 60 to 10 nm, and the roughness after a cycle of polishing with MRF takes 1.5 h. The roughness decreases from 4.157 to 0.851 nm in a 1.41 mm × 1.06 mm area scope. The periodical tool mark of the mirror surface after SPDT is swiped out by MRF. The surface morphology and XRD results manifest that the surface after MRF is clean and smooth.

The experiment results and theoretical analysis of this work prove that MRF can satisfy the ultra-precision fabrication of the NiP layer on the metal mirror by adopting suitable MR polishing fluid and parameters. The results also provide processing technical support for the application of metal in the visible and ultraviolet systems. To expand the application of metal mirrors to an ultraviolet or extreme ultraviolet system, optimization work should be carried out in the future to obtain the optimal parameters by combining more polishing parameters, such as flow, magnetic field, and depth of penetration. The powders' (iron particles or abrasives) modification is also under research to improve the processing efficiency and reduce the surface roughness further.

Funding. National Natural Science Foundation of China (NSFC) (51775531, 61605202).

REFERENCES

1. R. Steinkopf, A. Gebhardt, S. Scheiding, M. Rohde, O. Stenzel, S. Glied, V. Giggel, H. Löscher, G. Ullrich, P. Rucks, A. Duparre, S. Risse, R. Eberhardt, and A. Tünnermann, "Metal mirrors with excellent figure and roughness," *Proc. SPIE* **7102**, 71020C (2008).

2. S. Risse, A. Gebhardt, C. Damm, T. Peschel, W. Stöckl, T. Feigl, S. Kirschstein, R. Eberhardt, N. Kaiser, and A. Tünnermann, "Novel TMA telescope based on ultra precise metal mirrors," *Proc. SPIE* **7010**, 701016 (2010).
3. Y. C. Cheng, W. Y. Hsu, C. H. Kuo, K. A. E. Hossein, and T. Otieno, "Investigation of rapidly solidified aluminum by using diamond turning and a magnetorheological finishing process," *Proc. SPIE* **9575**, 957519 (2015).
4. S. Kim, S. Chang, S. Pak, K. J. Lee, B. Jeong, G. J. Lee, G. H. Kim, S. K. Shin, and S. M. Yoo, "Fabrication of electroless nickel plated aluminum freeform mirror for an infrared off-axis telescope," *Appl. Opt.* **54**, 10137–10144 (2015).
5. A. Beaucamp and R. R. Freeman, "Fluid jet and bonnet polishing of optical moulds for application from visible to x-ray," *Proc. SPIE* **8126**, 81260U (2011).
6. A. Beaucamp, R. Freeman, and R. Morton, "Removal of diamond-turning signatures on x-ray mandrels and metal optics by fluid-jet polishing," *Proc. SPIE* **7018**, 701835 (2008).
7. M. Beier, S. Scheiding, and A. Gebhardt, "Fabrication of high precision metallic freeform mirrors with magnetorheological finishing (MRF)," *Proc. SPIE* **8884**, 88840S (2013).
8. J. Reimers, A. Bauer, K. P. Thompson, and J. P. Rolland, "Freeform spectrometer enabling increased compactness," *Light Sci. Appl.* **6**, e17026 (2017).
9. C. Supranowitz, C. Hall, P. Dumas, and B. Hallock, "Improving surface figure and microroughness of IR materials and diamond turned surfaces with magnetorheological finishing (MRF)," *Proc. SPIE* **6545**, 65450S (2007).
10. M. W. Jeon, S. Hyun, B. J. Jeong, I.-J. Kim, and G.-H. Kim, "Removal of diamond turning marks with magneto-rheological finishing," *Proc. SPIE* **10371**, 103710V (2017).
11. P. Dumas, D. Golini, and M. Tricard, "Improve figure and finish of diamond turned surfaces with magneto-rheological finishing (MRF)," *Proc. SPIE* **5786**, 296–304 (2005).
12. D. Golini, W. I. Kordonski, P. Dumas, and S. J. Hogan, "Magnetorheological finishing (MRF) in commercial precision optics manufacturing," *Proc. SPIE* **3782**, 80–91 (1999).
13. Y. Bai, F. Zhang, W. J. Deng, L. X. Li, L. G. Zheng, and X. J. Zhang, "Preparation of magnetorheological polishing fluid and its polishing stability," *Acta Opt. Sinica* **34**, 0416001 (2014).
14. Y. Bai, L. X. Li, D. L. Xue, and X. J. Zhang, "Rapid fabrication of silicon modification layer on silicon carbide substrate," *Appl. Opt.* **55**, 5814–5820 (2016).
15. D. C. Harris, "History of magnetorheological finishing," *Proc. SPIE* **8016**, 80160N (2011).
16. V. I. Kordonski and S. R. Gorodkin, "Magnetorheological polishing of optical surfaces," *J. Opt. Technol.* **79**, 588–598 (2012).
17. J. Kinast, E. Hilpert, N. Lange, A. Gebhardt, R. R. Rohloff, S. Risse, R. Eberhardt, and A. Tünnermann, "Minimizing the bimetallic bending for cryogenic metal optics based on electroless nickel," *Proc. SPIE* **9151**, 915136 (2014).
18. S. N. Shafir, H. J. Romanofsky, M. Sharlinski, M. Wang, C. Miao, S. Salzman, T. Chartier, J. Mici, J. C. Lambropoulos, R. Shen, H. Yang, and S. D. Jacobs, "Zirconia-coated carbonyl iron particle based magnetorheological fluid for polishing optical glasses and ceramics," *Appl. Opt.* **48**, 6797–6810 (2009).
19. S. Salzman, H. J. Romanofsky, L. J. Giannechini, S. D. Jacobs, and J. C. Lambropoulos, "Magnetorheological finishing of chemical vapor deposited zinc sulfide via chemically and mechanically modified fluids," *Appl. Opt.* **55**, 1481–1489 (2016).
20. M. Schinhaerl, E. Pitschke, A. Geiss, R. Rascher, P. Sperber, R. Stamp, L. Smith, and G. Smith, "Comparison of different magnetorheological polishing fluids," *Proc. SPIE* **5965**, 596528 (2005).
21. W. W. You, X. Q. Peng, and Y. F. Dai, "MR fluids for finishing use," *Opt. Precis. Eng.* **12**, 330–334 (2004).
22. X. Q. Peng, F. F. Jiao, H. F. Cheng, G. Tie, F. Shi, and H. Hu, "Novel magnetorheological figuring of KDP crystal," *Chin. Opt. Lett.* **9**, 102201 (2011).
23. W. Kordonski and S. Gorodkin, "Material removal in magnetorheological finishing of optics," *Appl. Opt.* **50**, 1984–1994 (2011).
24. S. F. Ang, T. Scholz, A. Klocke, and G. A. Schneider, "Determination of elastic/plastic transition of human enamel by nanoindentation," *Dent. Mater.* **25**, 1403–1410 (2009).