Experimental Study on Chemical Polishing of Laser Powder Bed Fusion-Based Inconel 718 Features

B. Srinivas, M. M. Basha, and M. R. Sankar

1 Introduction

In additive manufacturing (AM), the material is added layer by layer rather than removing it like most unconventional machining methods, and complex near net shape parts can be produced using AM technologies [1]. The additive manufactured parts surfaces are generally rough and need a post-processing process, i.e., finishing processes like shot peening, vibratory finishing, laser polishing, chemical polishing, electrolytic polishing, etc. [2, 3]. This study aims to study the chemical polishing of Inconel alloy manufactured by additive manufacturing technique of selective laser melting (SLM). SLM is a powder-based additive manufacturing process in which powder is used as raw material to produce metallic components usually under vacuum or inert atmosphere [2, 3]. Chemical polishing is applicable for both conventional as well as additive manufacturing. The most incurring problem of the additive manufacturing process is loose powder particles that are partially sintered particles that stick on the surface. This problem needs to be addressed by a feasible and acceptable post-processing technique. Here, chemical polishing is one of the techniques which serves the purpose [5]. Researchers tried different iterations to get the optimal chemical composition, chemical concentration, optimal polishing time, and corresponding dynamic action [6]. Due to surface irregularities, there is a chance to crack formation during functionality. If it is finished using chemical polishing, then cracks are closed,

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and fatigue strength is increased [3, 7]. The chemical polishing technique is a broad area for research at present and in near future.

Inconel 718 is a Ni-based superalloy that has excellent properties. It has to retain mechanical stability within the range of 253–650 °C, good fatigue strength, corrosion resistance, resistance to oxidation (chromium and molybdenum presence contributes to oxidation resistance), and creep resistance [8]. These are the properties useful in hostile environments or applications like aircraft engines, jet engines, and steam turbine power plants. If parts are manufactured by a conventional method such as casting, then the part needs to be post-processed for improvement of surface morphology and dimensional accuracy. It involves tool wear in conventional machining while finishing Ni-based superalloys. Due to Inconel 718 having good weldability, powder bed fusion could be the prospective candidate for making Inconel 718 parts. To date, no effective chemical polishing solution has been found for nickel-based superalloys. Semi-welded particles are formed in the heat-affected zone by sintering of the loose powder particles nearby melt pool which depends on the energy density of the laser scanning which is related to temperature [9].

2 Chemical Polishing

The mechanism involves in the chemical polishing process or bath is the formation of a passive layer on the surface of the part; generally, the formed layer dissolves in the chemical polishing solution [10]. If it does not dissolve in the solution, then a complexing agent is used to make it soluble in the polishing solution even with a small amount of agitation using an ultrasonic cleaner or magnetic stirrer to increase the polishing rate. The removal of material from the surface of the valleys is less as compared to peaks because of deficiency of water in the pits which was explained clearly in viscous film theory [11]. Figure 1 represents the overall chemical polishing process.

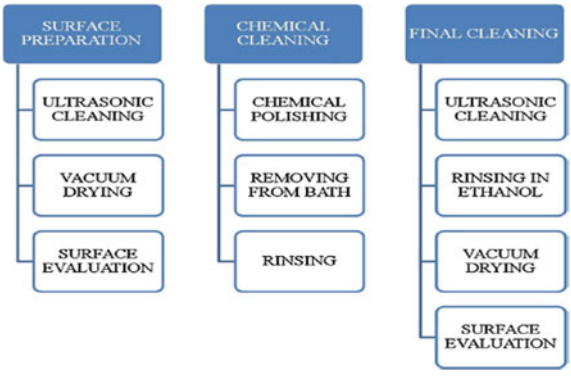

Fig. 1 Chemical polishing process [6]

Before polishing, additive manufactured surface needs to be prepared on which loosely bond powder particles are removed using ultrasonic cleaner, and it needs to be dried using a vacuum drier, and then, surface morphology is evaluated. After that actual cleaning is done, i.e., surface is immersed in a chemical bath where polishing takes place removal of extra powder particles from the surface is done, then removal of the part from the bath and rinse it with clean water to ensure removal of any chemical residue. Finally, after the chemical bath, final cleaning is done, and surface evaluation is done [6, 12].

Generally, a chemical bath contains two agents. They are an oxidizing agent and complexing agent. After immersion of a part in the bath, the immediate reaction between bath and surface is the oxidization–reduction process. Oxidizing agent of the solution oxidizes the surface material. Oxidization product is usually not soluble in the polishing solution and adheres to the surface. If the oxidization product is not soluble in the polishing solution, then, the complexing agent reacts with the oxidization product and makes it soluble in the solution. Examples of an oxidizing agent are nitric acid (HNO_3), hydrogen peroxide (H_2O_2), and for a complexing agent for titanium, the sample is hydrofluoric acid (HF). Most often, water is used to dilute the solution [13].

Anodic reaction: where the surface of solid is dissolved in oxidizing solution and surface atom (A) becomes positive by losing an electron.

$$A \rightarrow A^{+} + e^{-}$$

Cathodic reaction: where the oxidizing agent (X) diffuses to the surface and gains the electron.

$$X + e^{-} \rightarrow X^{-}$$

Therefore, the overall reaction would be

$$A + X \rightleftharpoons AX$$

The surface atom can be removed if the compound AX is soluble in the chemical polishing solution otherwise complexing agent should be used to make compound AX soluble. In the chemical polishing of a semiconductor, the polishing constituents react with each other. Aqua regia (HNO_3 + 3HCl) is an example of it and forms chlorine which is not desirable since it contributes to ozone layer depletion. Neda Mohammadian et al. [9] worked on SLM built 3D printed Inconel 625 components to enhance the interior surface quality of tubular structure used in the aerospace industry by reducing the surface roughness, i.e., removing semi-welded particles adhered on powder-based additive manufactured Inconel 625 component surface and to fix the composition of chemical solution with abrasives such that machinery should not be damaged using hybrid mechanism, i.e., with chemical–abrasive flow polishing. The chemical solution consisting of 50% HF + 50% HNO_3 was used

as a chemical solution in chemical assisted abrasive flow finishing technique, since it reduced the surface roughness considerably compared with other solutions like 30% HNO_3 + 10% H_2SO_4 + 10% H_3PO_4 + 50% acetic acid, 64.5% acetic acid (ice cooled) + 35% HNO_3 + 0.5%HCl and 30% HF + 70% HNO_3. Finally, 40 vol.% HF + 40 vol.% HNO_3 + 20 vol.% H_2O (diluted) with abrasives alumina were selected to take care of the machinery. Various polishing techniques were compared in which static chemical polishing achieved R_a = 14.2 μm from 17.2 μm. Huang et al. [14] polished electrochemically Inconel 718 with rotating disk electrodes using $HClO_4$ and CH_3COOH solution with various concentrations. By using 10–20 vol.% $HClO_4$ observed leveled surface without brightening, 30–40 vol.% $HClO_4$ observed both leveled and brightened surface and with 50 vol.% or more $HClO_4$ got matt and gray surface by the mechanism of precipitation of salt film on the surface, and simultaneously, the corrosion resistance was enhanced.

3 Materials and Methods

The 3D printed EOS Inconel 718 sample having a density of 8.15 g/cc was used for experimentation. The sample was the rod of dimensions of 15 mm diameter and 150 mm length which were measured by Vernier Calipers. The powder particles and sample are shown in Fig. 2a, b respectively.

Enough samples were cut using WEDM for various experimentation like microstructural observation, on longitudinal cross-section (section along the longitudinal axis) and on transverse cross-section (a circular section which is perpendicular to the longitudinal axis) concerning building direction which is parallel to the longitudinal axis and for optimizing the constituents of chemical polishing solution, RPM of magnetic stirrer and polishing time thereby improving surface quality. The elemental composition of EOS Inconel 718 was known by scanning electron microscope by point EDS. And elemental composition is given in Table 1.

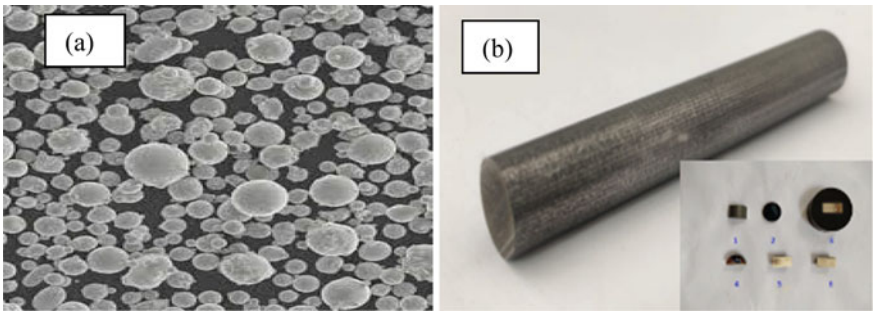

Fig. 2 **a** Inconel 718 particles used in laser powder bed fusion and **b** 3D printed Inconel 718 sample

Table 1 Elemental composition of 3D printed Inconel 718

Element	Ni	Cr	Fe	Ti	Mo	Mn	Al	Ta	Co	Cu
Wt. %	41.08	20.14	16.96	3.64	5.92	1.88	2.2	1.81	2.06	3.44

Microstructures are observed by using optical microscopy and secondary electron microscopy for both longitudinal and transverse cross-sections. Three different types of etchants H_2SO_4 + methanol, ethanol + HCl + $FeCl_3$, and HF + HCl + HNO_3 were tried to optimize the constituents of the bath. Here, a sequence of steps was followed; average surface roughness values were noted before chemical polishing followed by samples were cleaned properly using an ultrasonic cleaner, and then immersed in a chemical bath to allow for chemical polishing. After chemical polishing, samples were cleaned using ultrasonic cleaner, and surface roughness values were measured. For all the samples which are used for experimentation, weights were calculated before and after chemical polishing using digital weighing balance and weight loss % was calculated.

4 Results and Discussion

4.1 Microstructural Studies

The elemental composition of the sample before chemical polishing was known by scanning electron microscope (SEM) with point EDAX. The SEM image with focused point and corresponding SEM graph with a number of counts vs energy are shown in Fig. 3 are shown, respectively, and elemental composition was identified.

The WEDM cut samples longitudinal cross-section and transverse cross-section were subjected to manual polishing using emery papers of grit sizes in the sequence of 120, 240, 400, 600, 800, 1000 and velvet cloth polishing with colloidal silica solution on double disk manual polishing machine. Afterward, these samples were rinsed with distilled water and cleaned with CH_3OH using cotton and again rinsed

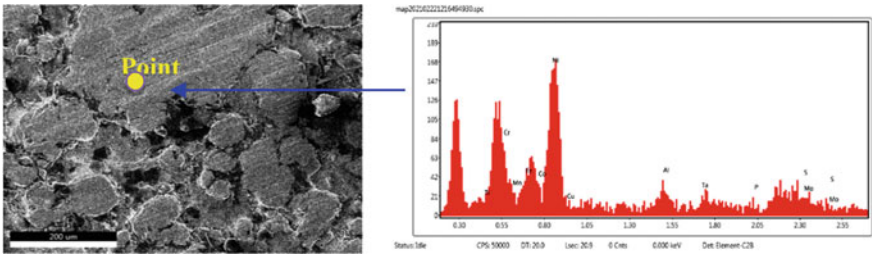

Fig. 3 Surface morphology of the 3D printed Inconel 718 surface before chemical polishing and its energy dispersive spectroscopy pattern

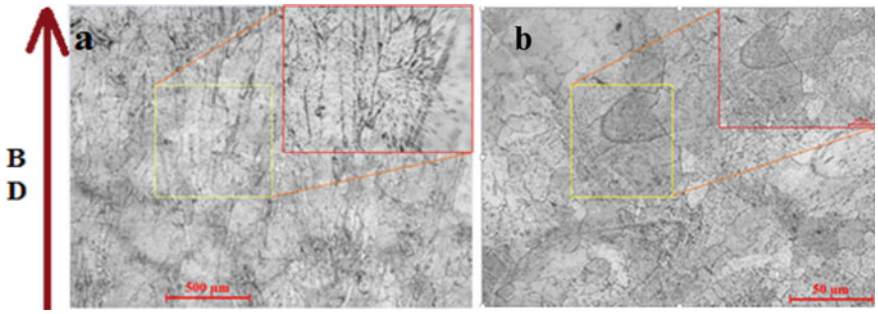

Fig. 4 Optical micrographs of **a** longitudinal and **b** transverse cross-sections. BD stands for build direction which was along the longitudinal axis of the rod

with distilled water, then dried the samples using a heating gun. The etchant prepared based on literature Apparao et al. [15] of 50% HCl, 10% HNO_3, 2% HF, and 38% of distilled water, in the same proportion of 5 ml HCl, 1 ml HNO_3, 0.2 ml HF, and 3.8 ml distilled water and allowed to react for 40 s then rinsed with distilled water and dried. The etched samples were sent to the optical microscope and therefore observed microstructures for both longitudinal and transverse cross-sections as shown in Fig. 4a, b respectively.

4.2 Chemical Polishing Experiments on Inconel 718

The average surface roughness (R_a) of the sample before chemical polishing was 5.786 µm. For chemical polishing, few trails with three different polishing solutions were performed. The selection of chemical polishing solution was taken based on a few factors like the constituent which controls etching and acts as solvent, i.e., methanol, etching and polishing constituent, i.e., H_2SO_4. The chemical polishing solution was 20 vol.% H_2SO_4 + methanol was tried with two different samples at different polishing times 60 and 90 min using dynamic action of magnetic stirrer at 250 RPM, and the average surface roughness values 7.1 and 7.452 µm, respectively, were observed after chemical polishing. A magnetic stirrer with 250 rpm was used for polishing, and it makes the surface an active site and leads to rigorous etching locally as shown in Fig. 5. However, there are no pits observed because the H_2SO_4 acts as a viscous layer over the surface and promotes the local etching. So, higher surface roughness values 7.1 and 7.452 µm were observed which is beyond the initial surface roughness (initial R_a = 5.78 µm). Finally, no considerable results were obtained. Figure 6a, b shown are before and after chemical, polishing using 20 vol.% H_2SO_4 + methanol with 250 RPM and 90 min polishing time and the corresponding final surface roughness profile over the evaluation length with final R_a = 7.452 µm as shown in Fig. 6c.

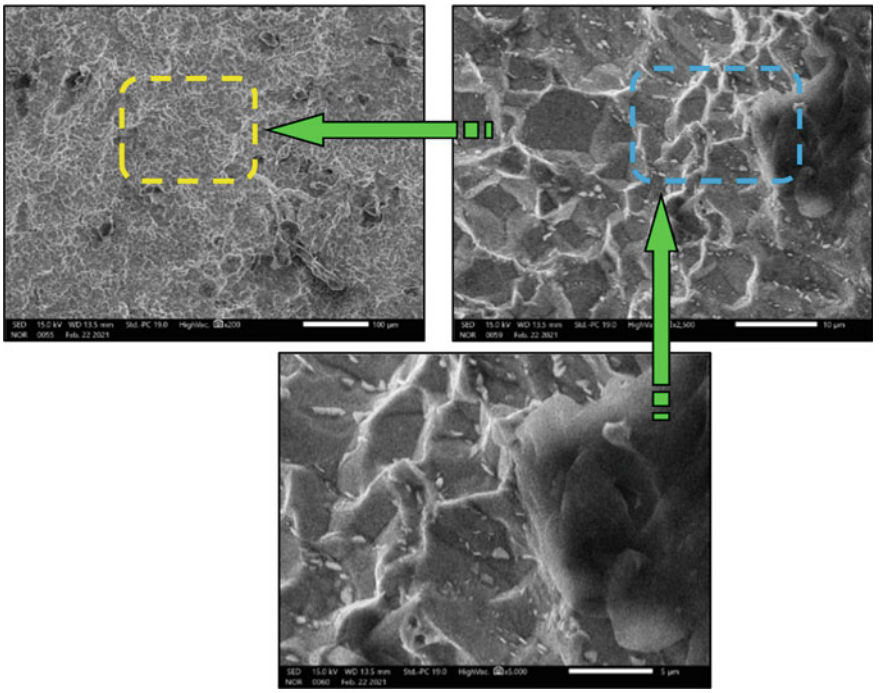

Fig. 5 Surface morphology after chemical polishing using 20 vol.% H_2SO_4 + methanol with 250 RPM and 90 min polishing time

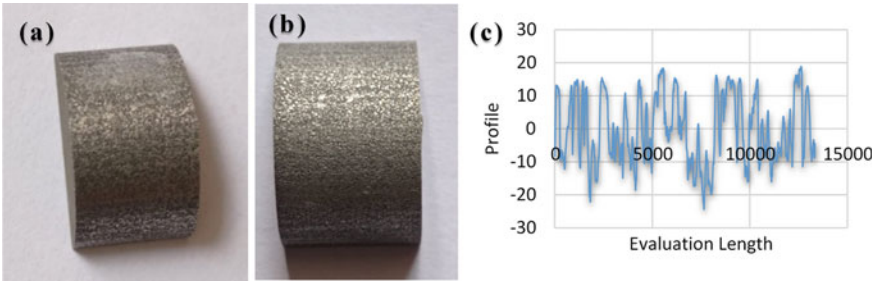

Fig. 6 **a** Before, **b** after chemical polishing, and **c** final surface roughness after chemical polishing

The solution consisting of ethanol (solvent and controls etching), HCl (etches), and $FeCl_3$ (brightening) was considered as another trial and tried to optimize the polishing bath under static conditions without a magnetic stirrer. By varying the ethanol from 10 to 18 ml (in volume) and keeping HCl (2.5 ml) and $FeCl_3$ (0.6 g) are constant and corresponding % weight loss also calculated as shown in Table 2.

While the increasing volume of ethanol to the polishing solution, more oxygen is generated and leads to localized pitting, so surface roughness values were increased.

Table 2 Variation of ethanol in the bath and time under static conditions

Ethanol (ml)	HCl (ml)	$FeCl_3$ (g)	Time (min)	R_a (μm)	%wt. loss
18	2.5	0.6	90	6.39	0.749
10	2.5	0.6	90	5.756	0.399
12	2.5	0.6	90	4.194	0.439

HCl is not acting as viscous, so it can also indirectly influence the generating pits. More pits are generated without showing the difference between the strength of grain and grain boundary as shown in Fig. 7. The solution consists of 12 ml ethanol, 2.5 ml HCl, and 0.6 g with polishing time of 90 min under static conditions without magnetic stirrer gave the better results, i.e., R_a from 5.786 to 4.194 μm, and the respective sample is shown in Fig. 8b, and corresponding final surface roughness profile over evaluation length is shown in Fig. 8c.

The polishing bath consists of HF, HCl, and HNO_3 were chosen based on HF acts as a complexing agent (which dissolves the oxide layer), HCl (which participates in etching), and HNO_3 acts as an oxidizing agent as well as participates in polishing. The polishing bath was optimized for better results by keeping HF (10 ml) and HCl

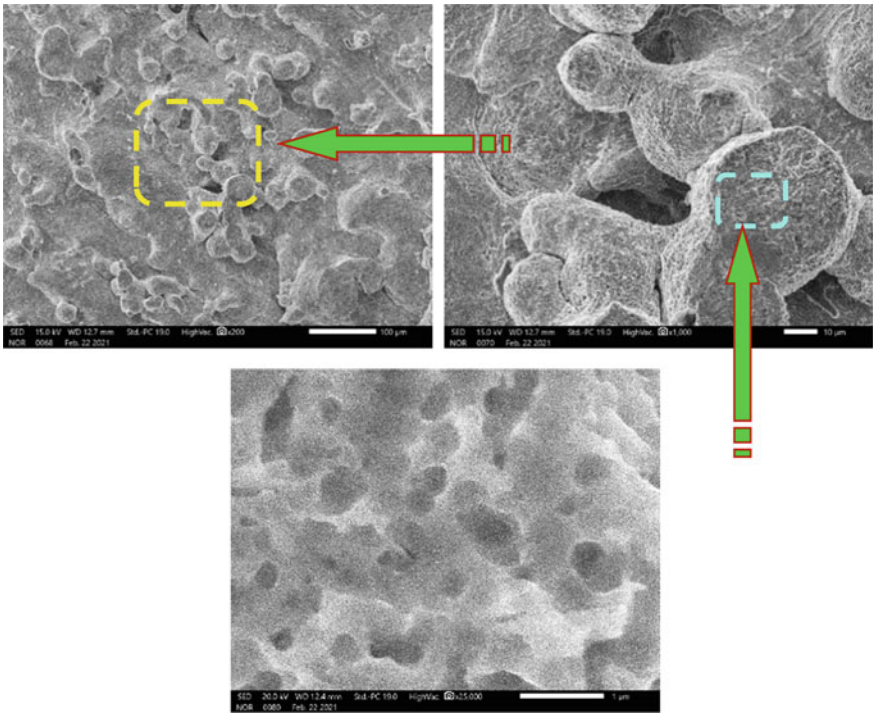

Fig. 7 SEM micrographs for polishing of sample under the static conditions 18 ml ethanol, 2.5 ml HCl, 0.6 $FeCl_3$ (g), 90 min time ($R_a = 6.39$ μm)

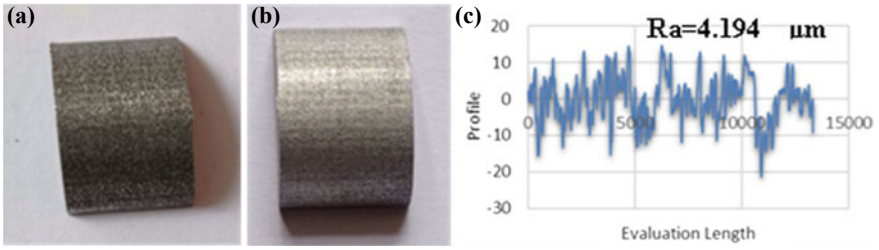

Fig. 8 **a** Before, **b** after chemical polishing, and **c** final surface roughness profile after polishing

Table 3 Variation of HNO_3 in the bath under static conditions

HF (ml)	HNO_3 (ml)	HCl (ml)	Time (min)	R_a (µm)	%wt. loss
10	10	5	120	6.81	0.321
10	9.8	5	120	7.577	0.270
10	9.5	5	120	8.411	0.299
10	9	5	120	4.072	0.304
10	8.5	5	120	5.96	0.380
10	8	5	120	6.05	0.296

(5 ml) as constants throughout the experimentation and varying HNO_3 from 8 to 10 ml with polishing time 120 min under static conditions without magnetic stirrer, and corresponding % wt loss values were calculated as shown in Table 3. Before and after chemical polishing, samples were cleaned in an ultrasonic cleaner for 10 min after polishing bath followed by ultrasonic cleaning average surface roughness values were measured.

Initially, HNO_3 acts as an etching solution, so material removal is increasing along with whatever the shape of the surface; therefore, R_a remained constant as shown in Fig. 9; then, it acts as an oxidizing agent, so material removal is decreasing up to a volume of 9.5 ml; consequently, good surface finish was observed. Afterward, pitting could be the reason for increasing the surface roughness values while keep on increasing the volume of HNO_3. The optical micrographs for final R_a values 4.072 and 6.81 µm are shown in Fig. 10a, b, respectively.

By using 10 ml HF, 10 ml HNO_3, and 5 ml HCl, with a polishing time of 120 min under static conditions without magnetic stirrer, more pits were observed leading to non-homogeneous etching in which grain boundaries appeared which confirms Inconel 718 has stronger grain boundaries than grains as shown in Fig. 11 with $R_a =$ 6.81 µm. Similarly, for other cases in which surface roughness values were increased, this could be the reason.

By using 10 ml HF, 9 ml HNO_3, and 5 ml HCl, with a polishing time of 120 min under static conditions without magnetic stirrer, only a few pits were observed leading to homogeneous etching as shown in Fig. 12 with $R_a = 4.072$ µm which is the better result among other baths. The samples which are shown in Fig. 13a, b are before

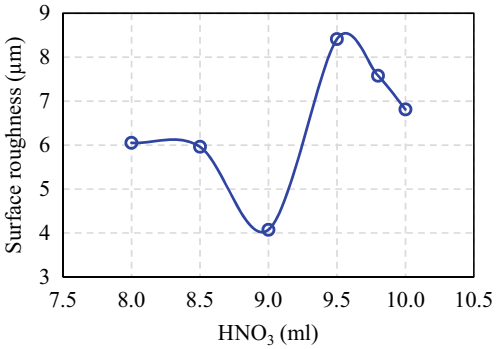

Fig. 9 Effect of HNO_3 in polishing solution at constant HF, HCl, and time

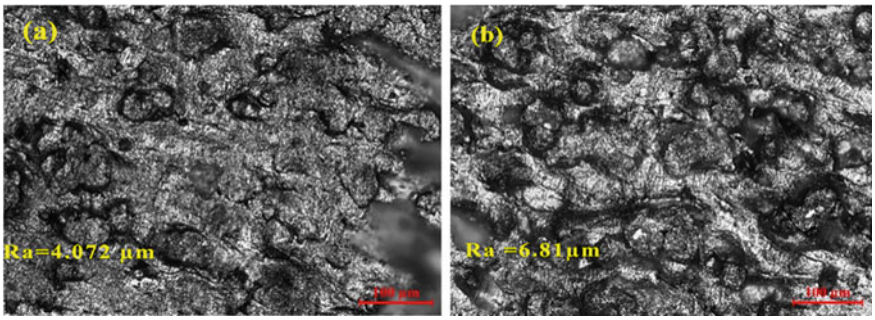

Fig. 10 Optical micrographs after chemical polishing at **a** 9 ml and **b** 10 ml HNO_3

polishing and after polishing using 10 ml HF, 9 ml HNO_3, and 5 ml HCl, with polishing time 120 min under static conditions, respectively, and Fig. 13c is the corresponding final surface roughness profile after chemical polishing with R_a = 4.072 µm.

5 Conclusions

In this paper, the conclusions are as follow:

- Etchant consisting of 50% HCl, 10% HNO_3, 2% HF, and 38% of distilled water was suitable etchant for microstructure observation.
- No remarkable results were observed using chemical polishing with dynamic action, i.e., with a magnetic stirrer at 250 rpm of bath consisting of 20 vol.% H_2SO_4 and methanol.
- The bath comprised of 12 ml ethanol + 2.5 ml HCl + 0.6 g $FeCl_3$ was the better polishing solution in trail 2 and reduction of surface roughness from R_a = 5.786 to 4.194 µm.

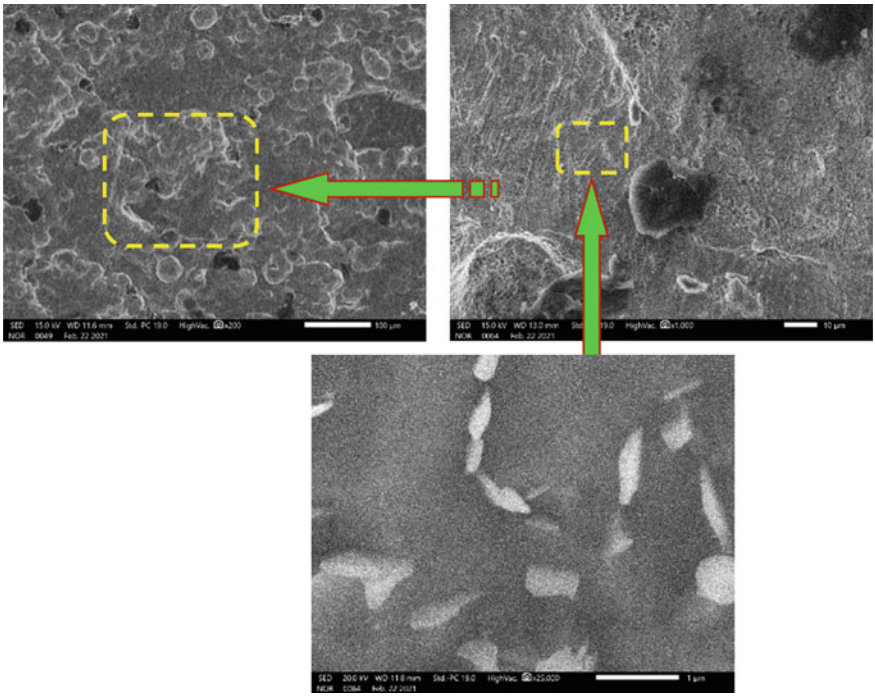

Fig. 11 Surface morphology after chemical polishing using 10 ml HF, 10 ml HNO_3, and 5 ml HCl, with polishing time 120 min under static conditions with more pits

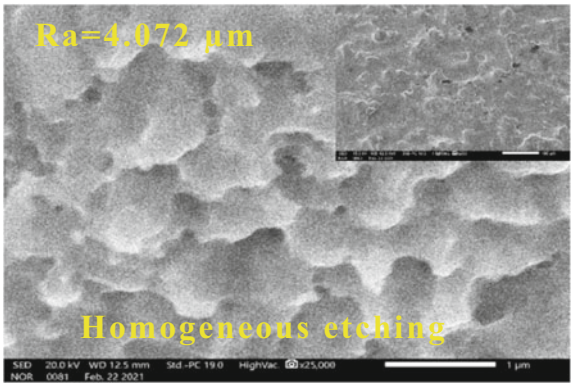

Fig. 12 Surface morphology after chemical polishing using 10 ml HF, 9 ml HNO_3, and 5 ml HCl, with polishing time 120 min under static conditions

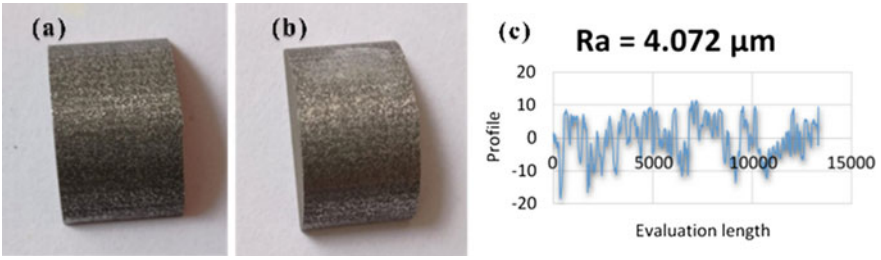

Fig. 13 **a** Before, **b** after chemical polishing, and **c** final roughness profile after chemical polishing

- The bath consists of 10 ml HF + 9 ml HNO_3 + 5 ml HCl was the better chemical polishing solution and reduction of surface roughness from R_a = 5.786 to 4.072 µm.

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