



# Recrystallization, Grain Growth, and Color Etching to Design an Artistic Microstructure in Cartridge Brass (C26000)

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## Abstract

This report details the creation of artistically engineered microstructural images for the 2021 International Metallographic Contest (IMC) and Exhibit. The concept for these images is to utilize purposeful recrystallization and grain growth to make designs in the microstructure. C26000 cartridge brass was the material used. The grains were initially grown to be large through annealing and then indentations were added. Recrystallization was induced, and the indentations were polished away to reveal the newly created grains in the designed pattern. The indentation spacing and load was determined by testing a range of loads and spacings for optimal parameters. The best conditions were determined to follow spacing between 80 and 100  $\mu\text{m}$  with a load of 1 kgf. The complex designs were engineered through AutoCAD to create CSV files which were input into a LECO automated indentation testing system (AMH55). Finally, three etchants from literature were investigated, Klemm's I, Klemm's II, and Klemm's III, with Klemm's II being chosen for its vibrant colors under polarized light. All of the investigations were combined to create several different images for submission to the International Metallographic Competition (IMC).

**Keywords** Recrystallization · Grain growth · Color etching · Localized cold work

## Introduction

Every year, the International Material Applications & Technologies (IMAT) conference hosts the International Metallographic Contest (IMC) and Exhibit where micrograph images can be submitted and judged. There are two categories for artistic micrographs, one in color and one in black and white [1]. Submissions for this contest in the past have often been discovered by chance. The goal of this research was to purposefully engineer a microstructure with an artistic design, image using light optical microscopy, and submit to the competition.

Single phase cartridge brass, C26000, an alloy composition of 70% copper and 30% zinc, was selected as the material for this project due to the striking twins and large grain size easily created by annealing a cold-worked sample, the

large range of contrast and colors that can be created by etching, and the availability of the material [2]. To purposefully engineer these microstructures, a Vickers microindenter was utilized to selectively cold work the C26000 cartridge brass in specific areas to create desired patterns. These specific cold-worked areas were annealed to create the intentional images from recrystallized grains. Color etchants were used to obtain contrast in micrographs. Colored etchants were chosen so that the image was created only through light optical microscopy (LOM) techniques and used no post processing or false coloring.

## Experimental Procedure

Samples of full hard (70% cold work) rolled cartridge brass were annealed at 850°C for 45 min to obtain large grains with an average grain size of 800  $\mu\text{m}$ . Samples were then mounted and polished to 1  $\mu\text{m}$  using conventional metallographic techniques.

The samples were indented using a manual Vickers microhardness tester. An array of indentations was

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created on each sample using forces of 1000, 500, 300, and 200 g-force (gf). Indentation spacings were varied from 3 mm to the point where they overlapped. Two lines of 10 indentations with equal spacings (150–75  $\mu\text{m}$ ) were created in order to attempt to produce a fine, continuous line of grains. Simple geometric shapes were also created using the same two spacings to attempt curved lines. This pattern of indentations was designed to test for the optimal spacings and load between indentations required to successfully recrystallize the cold-worked areas. The optimal indenter load and indentation spacing was determined to produce the most visible recrystallized grains with the cleanest overlap to create a continuous line. Samples were annealed at various temperatures from 450°–600°C for 15 min to recrystallize and determine a desired final grain size for visual preference. An anneal procedure of 525°C for 15 min was chosen.

Using the optimized parameters, Vickers microhardness indentations were performed using 1 kgf, 10 s dwell time, and spacing between 80 and 100  $\mu\text{m}$ . Samples were demounted and annealed at 525°C for 15 min. Samples were then mounted again and repolished taking care to stop coarse grinding steps once the Vickers microhardness indentations were no longer visible, and then fine polished through 1  $\mu\text{m}$  diamond polish followed by a 21-h vibratory polish with 0.05  $\mu\text{m}$  colloidal silica 138~140 V to 63.8 Hz frequency.

Samples were etched to reveal the microstructure and produce color etch effects. Klemm's reagents are recommended by Vander Voort to produce the most vibrant colors [3]. The three reagents were tested, Klemm's I, Klemm's II, and Klemm's III [4]. The three reagents contain the same constituents, sodium thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3$ ), potassium metabisulfite ( $\text{K}_2\text{S}_5\text{O}_5$ ), and deionized water, with varying compositions. Table 1 below shows the composition of each chemical prescribed by the American Society for Metals (ASM) handbook and recommended etch times.

To obtain the final colored images, the samples were then etched using Klemm's II reagent (50 mL saturated aqueous sodium thiosulfate mixed with 5 g potassium metabisulfite) [4, 5]. The optimized etch was determined to be 8 min in Klemm's II. Images were obtained with an Olympus DSX500 LOM with polarized light/differential interface contrast.

## Results and Discussion

The goal of this project was to create an artistic design using microstructural transformations with no photographic digital manipulation that would be pleasing to the viewer, and to leave the viewer initially questioning, "how did they do that". Our choice of C26000 cartridge brass was due to a large supply of material, over 10 years of recrystallization and grain growth data generated by laboratory classes at the Colorado School of Mines, and the beauty in the microstructure of recrystallized brass. We obtained our "canvas" by growing very large grains on the order of 800  $\mu\text{m}$  using an annealing treatment of 850°C for 45 min. Our as-received material was full hard cold-work brass sheet, so no further initial work hardening was needed to provide activation energy for recrystallization.

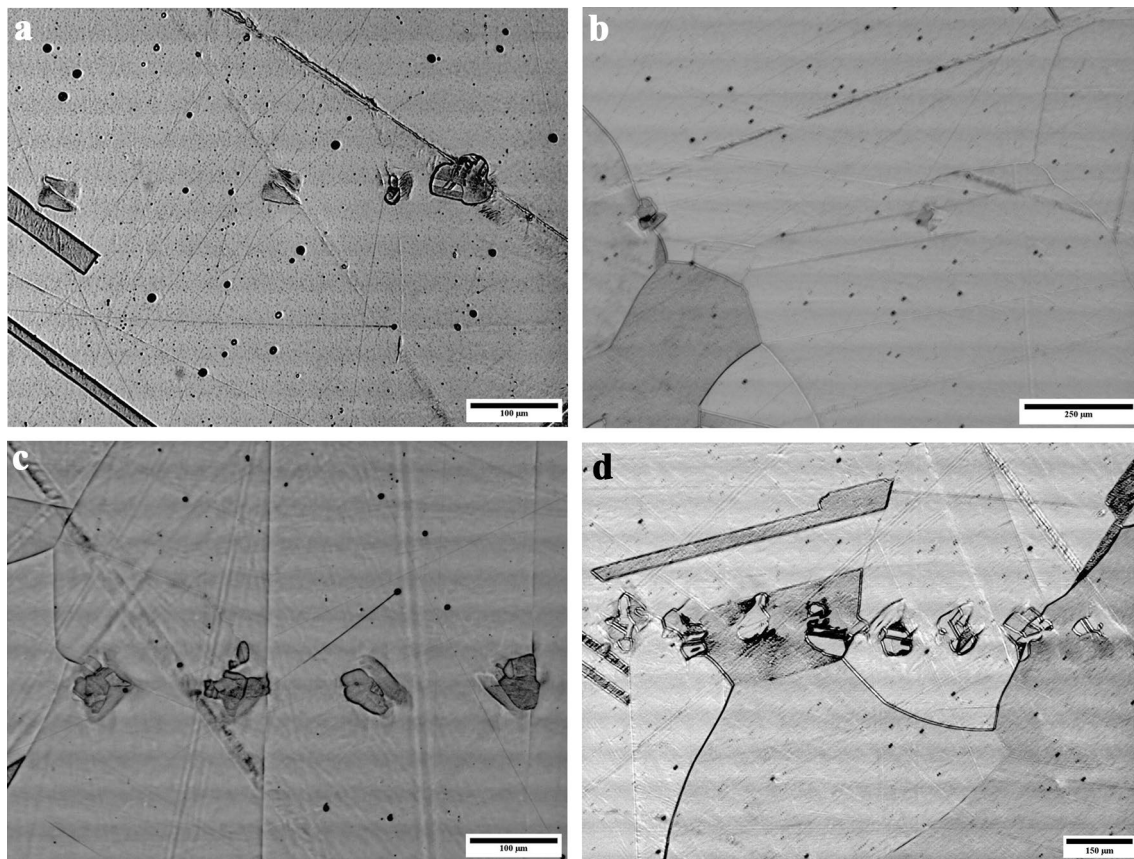
With our "canvas" we set out to create localized areas of deformation. We programmed a LECO AMH55 microhardness indenter so that we could import coordinates from an AutoCad converted image and then create a pattern of localized deformation. Several iterations were required to determine optimal load and spacings for our pattern. Figure 1 shows some results of iterating through indentation loads to produce sufficient cold work to initiate recrystallization.

As seen in Fig. 1, not every indentation produced significant recrystallization after annealing. Both the 200 gf and the 300 gf loads resulted in insufficient recrystallization after annealing. Figure 1c shows indentations made with equal spacing of ~150  $\mu\text{m}$  with a 500 gf load. While all indentations at 500 gf produced recrystallized grains, the grains that were produced did not overlap, so the optimal spacing was not achieved when a spacing of 150  $\mu\text{m}$  was used. The recrystallization for 500 gf could also be considered slightly less than optimal since the grain clusters are slightly smaller than desired. Figure 1d shows indentations at a spacing of ~150  $\mu\text{m}$  with a 1 kgf load. At the 1 kgf load, the recrystallized grains were observed to be an optimal and size, indicating the best force to utilize in later designs. However, the spacing of 150  $\mu\text{m}$  was still not close enough to produce a continuous line.

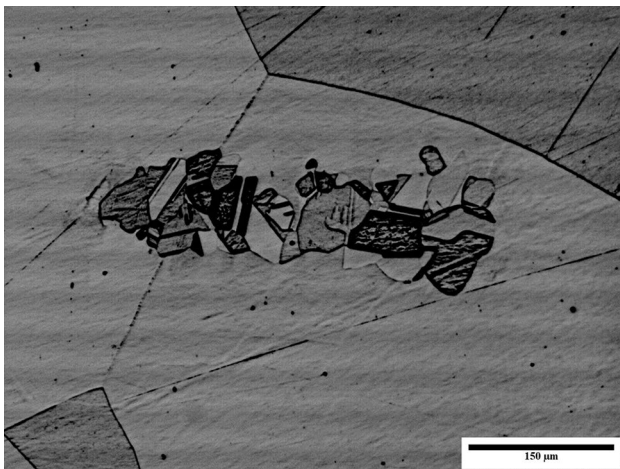
Figure 2 shows a continuous line of recrystallized grains created by indentations at a spacing of ~75  $\mu\text{m}$  at the 1 kgf load. Unlike the 150  $\mu\text{m}$  spacing, the 75  $\mu\text{m}$  spacing

**Table 1** Klemm's reagents compositions and etch times [4]

Etchant	Composition	Times
Klemm's I reagent	50 mL saturated $\text{Na}_2\text{S}_2\text{O}_3$ (stock solution), 1 g $\text{K}_2\text{S}_5\text{O}_5$	Up to 3 min
Klemm's II reagent	50 mL saturated $\text{Na}_2\text{S}_2\text{O}_3$ (stock solution), 5 g $\text{K}_2\text{S}_5\text{O}_5$	Up to 8 min
Klemm's III reagent	5 mL saturated $\text{Na}_2\text{S}_2\text{O}_3$ (stock solution), 20 g $\text{K}_2\text{S}_5\text{O}_5$ , 45 mL DI $\text{H}_2\text{O}$	Up to 8 min



**Fig. 1** Recrystallized grains: (a) exponentially decaying spacings at 200 gf, (b) 300 gf, (c) 500 gf with 150 µm spacing, (d) 1 kgf with 150 µm spacing



**Fig. 2** Recrystallization of 75 µm spacing at 1 kgf load

shows grain overlap. These grains, however, were deemed to be too clustered. For this reason, the optimal indentation spacing was determined to be between 80 and 100 µm. To test if this process could produce clean designs, the word “hi,” a square, and a smiley face were created using the

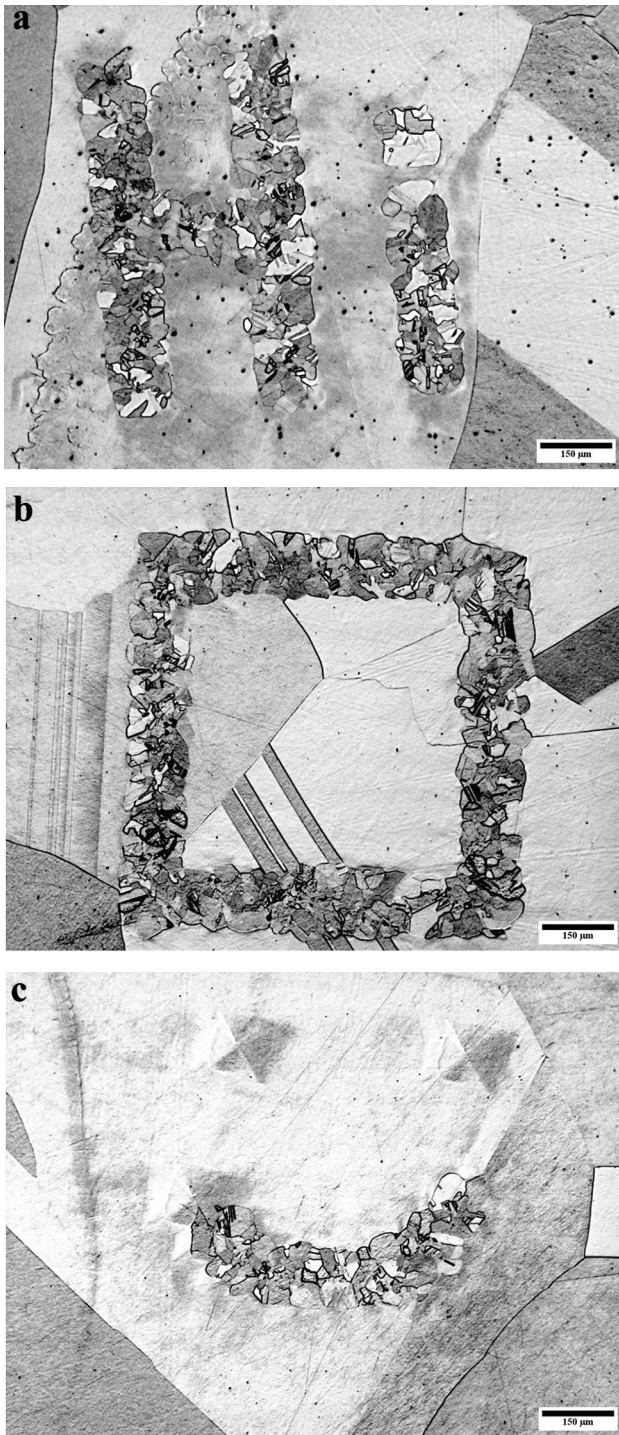
optimal load of 1 kgf and optimal spacing of between 80 and 100 µm, Fig 3a–c shows desired recrystallized grain continuity and density.

The etchants as described above in the procedure were carried out and the images showing the etchant colors can be seen below. All images were taken with polarized light using the Olympus DSX500 LOM with polarized light/differential interface contrast.

Samples were polished to 1 µm finish before being etched. According to the ASM handbook, the three Klemm’s reagents can be etched for a variety of different times. Figures 4, 5, and 6 below are the images acquired after applying the selected etchants to the brass samples at half the maximum time for each reagent (90 s for Klemm’s I and 4 min for Klemm’s II and III) and cleaned with soap and a cotton ball.

As seen in Fig. 4, Klemm’s I did not produce the desired vibrant colors. Klemm’s I was eliminated because of the lack of colors produced, as well as the lack of contrast between grain boundaries. A common problem between all of the etchants tested was a high density of scratches visible even after the 1 µm polish. To remedy the scratches, samples were polished to a 0.3 µm finish using an alumina solution. Figures 5 and 6 showed promise for producing a desired result.



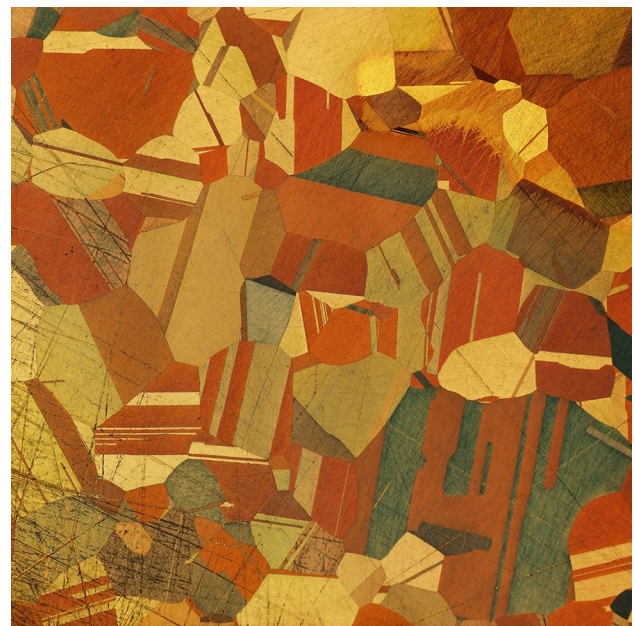


**Fig. 3** Annealed samples showing successful results with 1 kgf and 80–100 µm spacings after completed process. **(a)** shows the word “Hi”, **(b)** shows a square, and **(c)** shows a smiley face

Figure 7 shows a micrograph etched with Klemm’s II for the fully recommended time of 8 min after polishing to a 0.3 µm finish.



**Fig. 4** Klemm’s I applied for 90 s imaged with polarized light



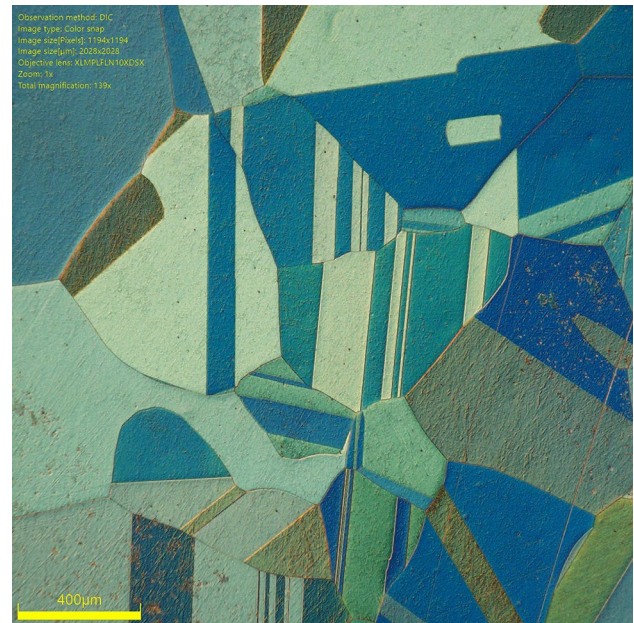
**Fig. 5** Klemm’s II applied for 4 min imaged with polarized light

Figure 7 shows that scratches were still visible after the 0.3 µm polishing. To further remedy the scratches, samples were further polished to a 0.05 µm finish using a colloidal silica solution on a vibratory polisher for 21 h. After etching, the samples were only rinsed with water to avoid further scratching from cotton ball cleaning. Figure 8 shows a

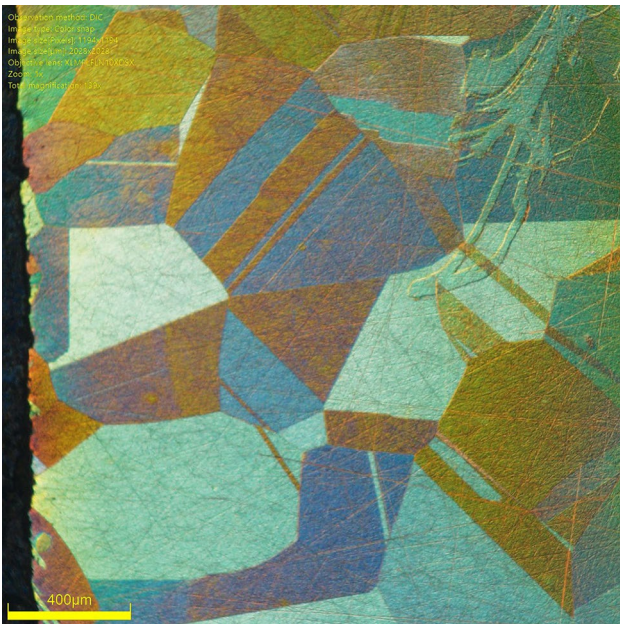




**Fig. 6** Klemm's III applied for 4 min image with polarized light



**Fig. 8** Klemm's II applied for 8 min after polishing to 0.05  $\mu\text{m}$  finish



**Fig. 7** Klemm's II applied for 8 min after polishing to 0.3  $\mu\text{m}$  finish



**Fig. 9** Klemm's III applied for 8 min after polishing to 0.05  $\mu\text{m}$  finish

micrograph etched with Klemm's II for 8 min after polishing to a 0.05  $\mu\text{m}$  finish.

Figure 8 shows significantly reduced scratching after etching. After testing, it was decided that Klemm's III was not optimal as there was a lack of reproducibility during etching. This may be due Klemm's III reagent not consistently staying in solution during etching and storage. Figure 9

shows a micrograph of the Klemm's III reagent applied for 8 min post colloidal silica polishing.

As seen in Fig. 9, the etching effect during the 8 min application of Klemm's III was determined to be undesirable. Due to the undesirable etch effect, and that the optimal colors were already achieved with Klemm's II reagent, Klemm's III testing was discontinued.



From all the etchant testing, it was determined that Klemm's II, applied for 8 min and rinsed with water, after polishing with colloidal silica returned the clearest and most colorful micrographs.

The designs used were created in AutoCAD. Once a design was sketched in AutoCAD, the divide function was used to divide lines into segments with endpoints with 80–100  $\mu\text{m}$  spacing. After all entities were cleared, leaving just the segment endpoints, the designs were exported as CSV files. The CSV files were then inputted into the AMH55 automated hardness testing system which indented the samples at the desired locations. The samples were then recrystallized, polished to a 0.05  $\mu\text{m}$  finish, and etched using Klemm's II reagent for 8 min. The images were then taken using the Olympus DSX500 LOM with polarized light/differential interface contrast. Figures 10, 11, 12 and 13 show the final images.

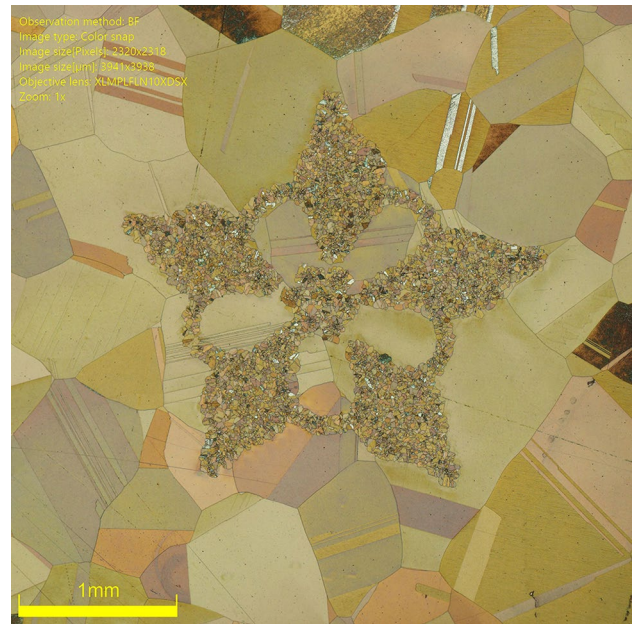
## Conclusion

Optimized processing conditions to engineer images into the microstructure of C26000 cartridge brass were determined. During annealing, a large grain matrix “canvas” was created in the microstructure of the brass. The AMH55 automated hardness testing system was utilized in conjunction with AutoCAD CSV files to create complex, artistic, and engineered designs. The sample experienced localized recrystallization and grain growth, and the indentations were polished to reveal the new grains in the desired pattern.



**Fig. 10** Columbine flower using bright field illumination

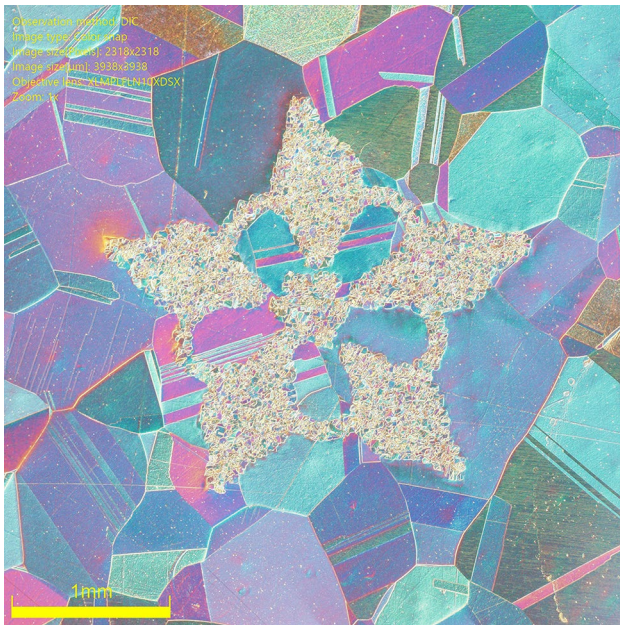
Through testing and experimentation, the optimal spacing was determined to be 80–100  $\mu\text{m}$  between indentations with a 1 kgf load. Three etchants, Klemm's I, Klemm's II, and Klemm's III, were investigated as potential etchants for the project. It was concluded that Klemm's II was the most suitable etchant for the desired results because of its vibrant



**Fig. 11** Columbine flower with shaded petals using bright field illumination



**Fig. 12** Columbine flower with shaded petals imaged with polarized light



**Fig. 13** Columbine flower with shaded petals imaged with polarized light

colors under polarized light. Using these processes, several images were created.

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