

# Surface study of irradiated sapphires from Phrae Province, Thailand using AFM

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**Abstract.** The irradiation is one of the gemstone enhancements for improving the gem quality. Typically, there are many varieties of irradiated gemstones in the gem market such as diamond, topaz, and sapphire. However, it is hard to identify the gemstones before and after irradiation. The aim of this study is to analyze the surface morphology for classifying the pristine and irradiated sapphires using atomic force microscope (AFM). In this study, the sapphire samples were collected from Phrae Province, Thailand. The samples were irradiated by high energy electron beam for a dose of ionizing radiation at 40,000 kGy. As the results, the surface morphology of pristine sapphires shows regular atomic arrangement, whereas, the surface morphology of irradiated sapphires shows the nano-channel observed by the 2D and 3D AFM images. The atomic step height and root mean square roughness have changed after irradiation due to the micro-structural defect on the sapphire surface. Therefore, this study is a frontier application for sapphire identification before and after irradiation.

## 1. Introduction

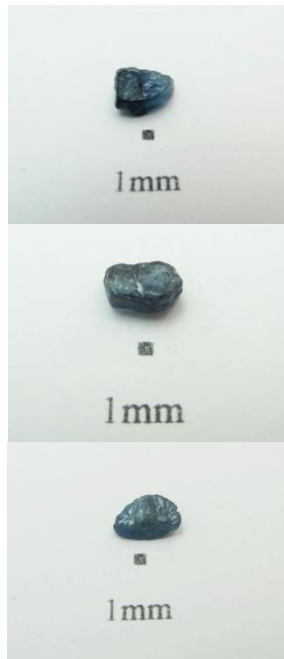
Sapphire is an alumina mineral consisting of  $\text{Al}_2\text{O}_3$  and trace elements such as Fe, Ti, Cr, etc [1]. The precious sapphires are valuable gems; however, it is quite difficult to find out in nature [2]. To improve their quality, therefore, there are many methods for gemstone enhancement including thermal enhancement, diffusion treatment, and irradiation [3]. For irradiation method, it has been applied for many types of gemstone such as diamond, topaz, and sapphire. There are many types of irradiation for gemstone enhancement such as gamma, neutron, and electron irradiation. Nowadays, it has been complicated to classify the pristine sapphire and irradiated one.

From previous works, the surface study of irradiated sapphire using atomic force microscope (AFM) was reported [4-7]. The electron beam generated by some swift heavy ions such as  $\text{Au}^{9+}$  have experimented in  $\text{Al}_2\text{O}_3$  film and the mean surface roughness was increased after irradiation [4]. Furthermore, the surface roughness of sapphires was changed by varying the dose effect corresponding to other research [6]. Thus, it is interesting to apply this technique on the natural sapphire in gem markets.

In this study, the surface morphology between pristine sapphire and irradiated one will be analyzed by AFM. The aim of this study is to identify the pristine sapphire and irradiated one based on their nano-surface morphology.

## 2. Experiment

In this study, the sapphire samples were collected from Phrae Province, Thailand for three samples. Then, the samples were irradiated using high energy electron beam at 40,000 kGy doses of ionizing radiation. The irradiation process was conducted by the high energy electron accelerator (MB 20-16 model) at the Gems Irradiation Center under Thailand Institute of Nuclear Technology (Public Organization). The irradiated sapphire samples were undergone the decay process to eliminate the residual irradiation before the AFM analysis. After irradiation process, the blue color of a sapphire samples was weakened as shown in figure 1.



**Figure 1a.** Pristine sapphires from Phrae Province, Thailand.

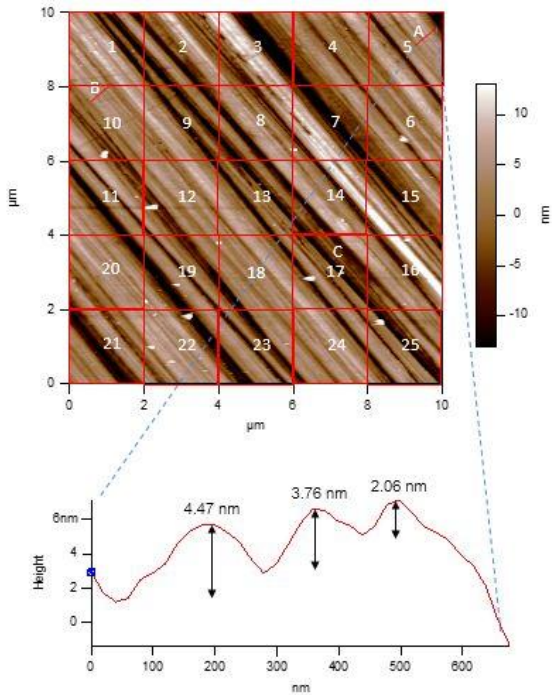


**Figure 1b.** Irradiated sapphires from Phrae Province, Thailand.

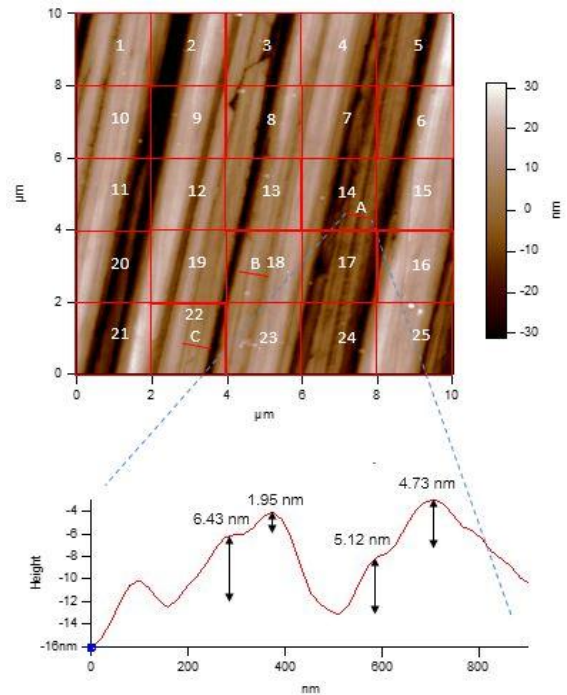
The surface morphology of pristine sapphires and irradiated sapphires was observed by atomic force microscope (AR MFP-3D model). Before AFM experiment, the samples were prepared by cleaning in 10% Decon solution followed by 1% NaOH and deionized water [8]. This experiment was set in the tapping mode (also known as non-contact mode) to measure the atomic force between the probed-tip and the sample surface. The 2D and 3D AFM images of the samples were analyzed by AFM Igor Pro 6.03A software.

## 3. Results and discussion

The 2D AFM images of a representative pristine sapphire from Phrae Province, Thailand comparing to irradiated one were focused on  $100 \mu\text{m}^2$  ( $10 \mu\text{m} \times 10 \mu\text{m}$ ) as shown in figure 2. The analyzing lines of each sample (such as line A, line B, and line C) were selected for calculating the average step height, besides, the whole area was regularly separated as the AFM mapping for investigating the root mean square roughness. It is hard to measure the step height and roughness of the samples in a similar position; however, those values could be reliable because the spreading of high energy electron beam could be affected covering the whole surface. The average step height and root mean square (RMS) roughness of the irradiated sapphire have significantly increased as shown in table 1. The result is quite consistent with other gemstone research, for example, the surface morphology of irradiated diamond [9].



**Figure 2a.** The 2D AFM image of a representative pristine sapphire from Phrae Province, Thailand.

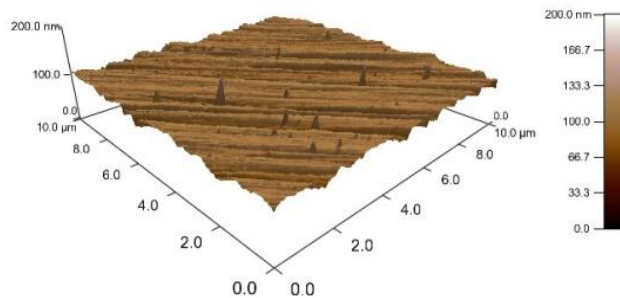


**Figure 2b.** The 2D AFM image of a representative irradiated sapphire from Phrae Province, Thailand.

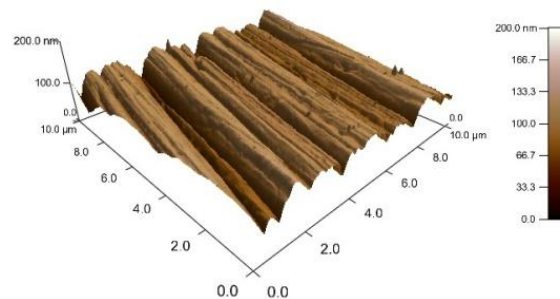
**Table 1.** The average step height and RMS roughness with a standard deviation (SD) of a representative pristine sapphire from Phrae Province, Thailand and irradiated one.

Sample	Average value (nm)	
	Step height	RMS roughness
Pristine sapphire	$2.86 \pm 1.38$	$6.42 \pm 1.26$
Irradiated sapphire	$3.92 \pm 1.40$	$15.25 \pm 3.21$

The 3D AFM images of a representative sapphire from Phrae Province, Thailand were shown in figure 3. For pristine sapphire, it is a regular atomic arrangement [7]. Besides, the surface morphology of irradiated sapphire was deeper nano-channel than the pristine one due to the effect of high energy electron beam which significantly corresponds to the increasing of atomic step height and RMS roughness.



**Figure 3a.** The 3D AFM image of a representative pristine sapphire from Phrae Province, Thailand.



**Figure 3b.** The 3D AFM image of a representative irradiated sapphire from Phrae Province, Thailand.

The increased RMS roughness of sapphire after irradiation could be related with the changes formation of nano-structure as shown as the 3D AFM images corresponding to other research [4, 5]. It is the result from the sputtering of the high energy electron beam on the sample surface as well as the high energy electron beam could be evaporated on the surface. It will be purposed that the micro-structural defect on the surface causing by the high energy electron beam could be evidenced to identify the pristine sapphire and the irradiated one. This study is a preliminary research to be a frontier standardized innovation for better gemstone identification unless an experience of gemologists. Besides, the time for irradiation is related to doses of irradiation; however, this study is focused only 40,000 kGy. For our ongoing research, it could be compared step height and RMS roughness of the samples varying different doses of irradiation.

#### 4. Conclusion

The surface morphology of natural sapphires from Phrae Province, Thailand has been modified by the effect of irradiation by high energy electron beam. Noticeably, it is an advantage of surface study applied to the gem identification before and after irradiated treatment.

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