

## Physiochemical properties of crystalline etch products for CR-39 track detector after $\alpha$ -particles irradiation

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### ABSTRACT

The effect of  $\alpha$ - particles from radiation source Am-241 with activity  $1\mu\text{Ci}$  on the nuclear track detector - NTD type CR-39, dimensions  $(28 \times 28 \times 1.2) \text{ mm}^3$  were measured. The measurements included the changes in physical and chemical properties for the solution of etch crystalline products for CR-39 after irradiation by  $\alpha$ -particles irradiation at two periodic times  $0.3 \text{ h}$  and  $23 \text{ h}$  for low and high doses respectively. The changes in organic compounds for etch products were measured by using of FTIR - spectroscopy and transmission electron microscope - TEM. FTIR - spectroscopy shown there was decrease in the values of transmission percent ratios -  $Tr$  of wave numbers  $1787$ ,  $2800$ ,  $839$  and  $1650 \text{ cm}^{-1}$  relative to wave number  $792 \text{ cm}^{-1}$  respectively with increase of irradiation time of  $\alpha$ -particles. The etch products after  $\alpha$ -particles irradiation of CR-39 detectors were change to low molecular weight organ compounds as a results of degradation bonds and may be formed compounds 2.2-oxy diethanol and 2-propen as the reaction products. Also, shown from transmission electron microscope -TEM there was increase in the degradation amount of etching organic compounds with increase of irradiation time of  $\alpha$ -particles. While the change in inorganic compounds for etch crystalline products after  $\alpha$ -particles irradiation of CR-39 detectors were describe by using of polarized optical microscope - POM , which shown from its images there was broken in crystalline layers to small crystal with increase of irradiation time . The physiochemical properties which measured by FTIR spectroscopy and transmission electron microscope-TEM for organic etching products were better than the analysis of inorganic crystalline etching products by Polarized microscope-PM .

**Keywords:** CR-39, nuclear track detector-NTD, crystalline etch products,  $\alpha$ - particles.

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### INTRODUCTION

CR-39 represent of one type of polymers which employed as a detectors in field of science and technology [ 1, 2 , 3] . one of these field was measurement the radiation effects for non-particle radiation as ionizing radiation[4] and particle radiation as ion beam , neutron and  $\alpha$ -particle[5] , and so named since named nuclear track detector-NTD[6,7] . The main strength of these detectors is that the damage produced by the ionizing particle can be enlarged through chemical etching [8] . These enlarged tracks, and physical properties can be viewed under the optical microscope and fourier transform infrared spectroscopy – FTIR [9,10] .

Nuclear track detector – NTD type CR-39 is one of the trade names of the family of Poly Allyl Diglycol Carbonate – PADC etch track detectors. When it is etched in sodium hydroxide – NaOH solution , a variety of inorganic and organic compounds are formed as the reaction products , which also effect on the thickness of CR-39 [11, 12]. Many products were formed after CR-39 etched in NaOH solution as poly allyl alcohol , 2.2-oxy diethanol [13] , allyl alcohol , isopropyl alcohol , sodium carbonate , sodium bicarbonate , and  $\text{CO}_3^{2-}$  ions [ 14 , 15 ] as well as crystalline products as nahcolite (  $\text{NaHCO}_3$  ) , natrite (  $\text{Na}_2\text{CO}_3 \cdot 7\text{H}_2\text{O}$  ) , thermonatrite (  $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$  ) , natron (  $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$  ) and trona (  $\text{Na}_2\text{CO}_3 \cdot \text{NaHCO}_3 \cdot 2\text{H}_2\text{O}$  )[14]. When the physical and chemical products of crystalline etch products is changes with type of radiation damage. So, in the present work , we examined the

properties of crystalline etch products of CR-39 after irradiation with  $\alpha$ - particles . The effect of  $\alpha$ - radiation on CR-39 detector through their crystalline etch products was measured by analysis of organic compounds of these products from using of FTIR – spectroscopy [9] and transmission electron microscope –TEM [16] , when the analysis of inorganic compounds [17] of these products from using of polarized microscope.

## MATERIALS AND METHODS

Three pieces sample of nuclear track detector-NTD type CR-39 was munched from Xuchang Tianhe Welding Products Co. , Ltd. Having dimensions ( 28 x 28 x 1.2 ) mm<sup>3</sup> . Two pieces samples of CR-39 were irradiated by  $\alpha$ -particle from Am-241 source with activity 1 $\mu$ Ci . One of these samples irradiated at 0.3 h ( for low radiation dose ) and another sample irradiated at 23h ( for high radiation dose) . Third sample of CR-39 was un-irradiated as a control .

Three sample above were dissolved in 200 ml of 6:0 M sodium hydroxide - NaOH solution contained in a 300 ml Pyrex beaker . The beaker was placed in a water bath maintained at 70.2 - 70.3°C and shacked for 70 h to prepare a super-saturated solution of the etch products . The separation of organic compounds solutions for CR-39 during etching process for three sample above were sampled as EC , ER<sub>1</sub> and ER<sub>2</sub> for un-irradiated , 0.3 h  $\alpha$ - irradiated and 23 h  $\alpha$ - irradiated respectively .

EC , ER<sub>1</sub> and ER<sub>2</sub> were separated in pure forms by solvent extraction with 50 ml diethyl ether with the aid of 250 ml separating funnel for three steps , then the organic extracting evaporated at room temperature up to 2 ml concentrate sample , and the FTIR spectra in the range (4000 – 400 ) Cm<sup>-1</sup> were recorded using NaCl sandwich cell on FTIR instrument , model - 8000 Shimadzu spectrophotometer . Separation of organic compounds was also analysis by transmission electron microscope - TEM , Model - Philips CM10 with an optimal operating voltage of 200 keV , to show the images of organic compounds after irradiation comparing with un-irradiated samples . The separation of inorganic compounds for EC , ER<sub>1</sub> and ER<sub>2</sub> by splitting the aqueous layer samples and left this layer to evaporate at room temperature to grow the formed inorganic products for 2 months , different needle crystalline for each sample were separated manually upon a glass microscope for image analysis by polarized optical microscope – POM , model Meiji MT9000. The texture of the compounds were observed using polarized light with crossed polarizer , the sample being prepared as a thin film sandwiched between a glass slide and a cover slip . A camera –Lumenera was installed on the polarizing microscope.

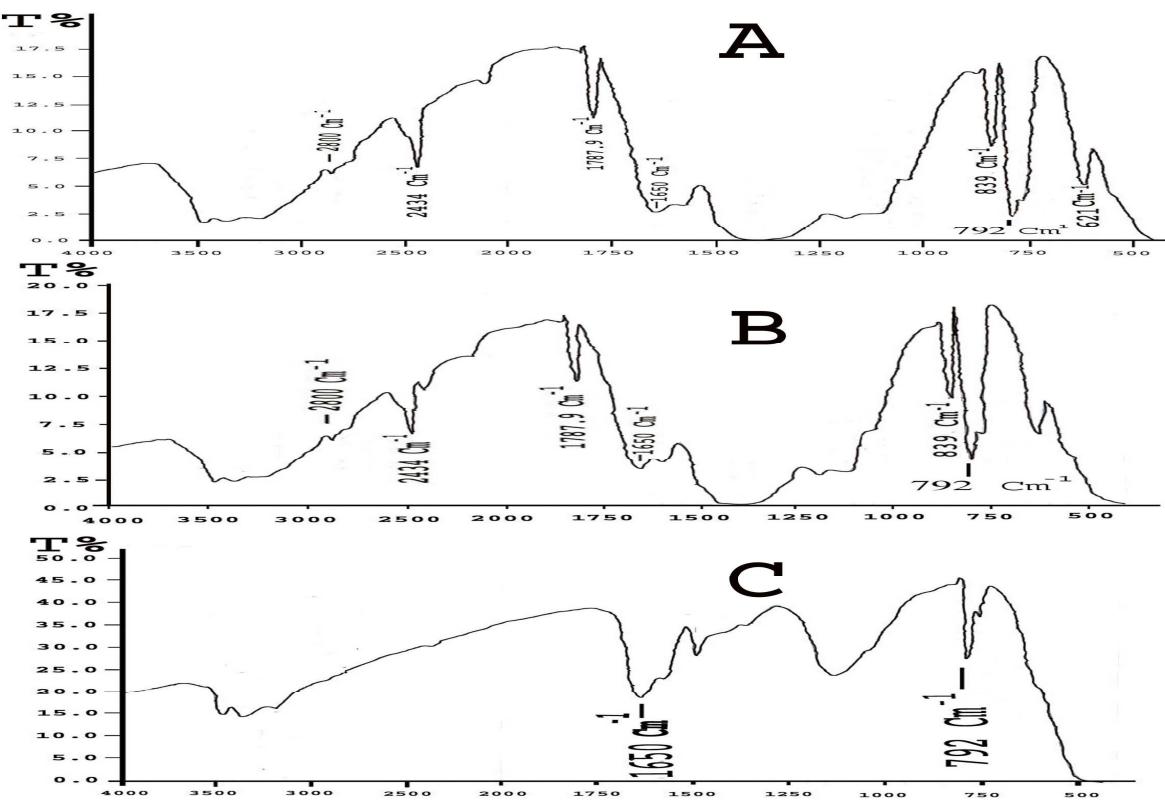
## RESULTS AND DISCUSSION

Figure 1 shown the FTIR spectrum at the wave number rang 4000-400 Cm<sup>-1</sup> for chemical etching products of EC , ER<sub>1</sub> and ER<sub>2</sub> samples for un-irradiated , 0.3 h irradiated and 23 h irradiated of CR-39 respectively . The FTIR spectrum for EC sample , show the presence of band at 1787.9 Cm<sup>-1</sup> due to the stretching of carbonyl group of ester and band at 839 Cm<sup>-1</sup> for out of plane bending , these two band also appeared for ER<sub>1</sub> while this two bands disappeared for ER<sub>2</sub>, which can attributed to the decomposition of compound ER<sub>2</sub> by the action of radiation and converted to another compounds (alcohol and ether) . Other bands like aliphatic C-H stretching at 2800 Cm<sup>-1</sup> and C = C stretching at 1650 Cm<sup>-1</sup> having small effect relative to wave numbers 1787.9 Cm<sup>-1</sup> and 839 Cm<sup>-1</sup> .

As we see from figures all the functional group decreases this due to as we said the decomposition of compound to lower molecular mass alkane during etching of CR-39, as shown in figure 2 .

The FTIR spectra show the appearance of OH stretching group for samples EC , ER<sub>1</sub> and ER<sub>2</sub> , because the reactant and the products contain hydroxyl group. Also from the spectra we have bands for carbonyl stretching at 1787.9 cm<sup>-1</sup> for sample EC and ER<sub>1</sub> and disappeared for sample ER<sub>2</sub> this can be explain as follows : sample EC contain two carbonyl groups, after radiation produce sample ER<sub>1</sub> which contain one carbonyl group, while sample ER<sub>2</sub> don't have carbonyl group as show in figure 2 .

Table 1 show the wave numbers and assignment for crystalline etch products for EC , ER<sub>1</sub> and ER<sub>2</sub> of CR-39 detector measured by FTIR spectroscopy , which shown the mains bonds still appear with increase of irradiation time at wave numbers 2800 , 1787 , 1650 , 839 Cm<sup>-1</sup> as well as to another wave numbers 3300 , 1402 , 1109 Cm<sup>-1</sup> .



**Fig. 1 :** Transmission percent –T% from FTIR – spectrum at wave number range 4000- 400 Cm<sup>-1</sup> for organic etching products of EC , ER<sub>1</sub> and ER<sub>2</sub> samples after  $\alpha$  –particle irradiated of CR-39 detector respectively . A: un-irradiated , B: 0.3 h irradiated , C: 23 h irradiated

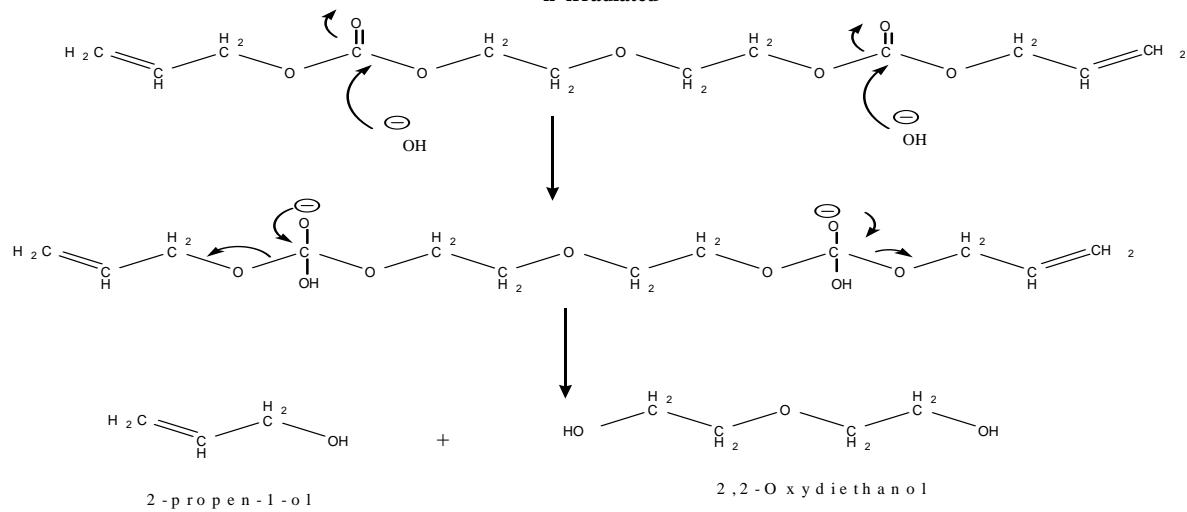


Fig. 2 : Scheme for the possible positions which are subjected to cleavage by the hydroxide ion [ 13 ].

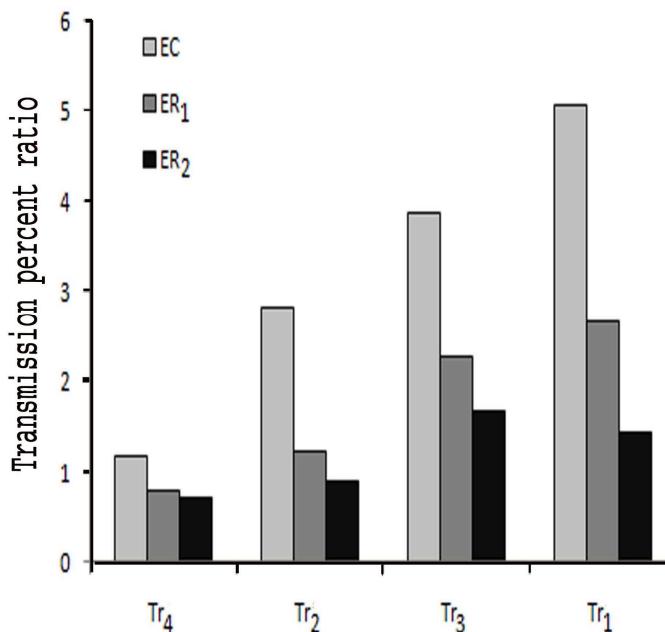
**Table 1 : Wave numbers – Cm<sup>-1</sup> and assignment crystalline etch products of for chemical etching products of CR-39 detector measured by FTIR spectroscopy as per Figure 1**

ESC (Cm <sup>-1</sup> )	ESR <sub>1</sub> (Cm <sup>-1</sup> )	ESR <sub>2</sub> (Cm <sup>-1</sup> )	Assignment
3300 3193	~ 3300 3192	3348 3190	OH stretching C=C-H stretching
2800 2761	2800 2761	~ 2860 -----	Symmetric CH <sub>2</sub> stretching Symmetric CH <sub>2</sub> stretching
1787.9	1787.9	-----	C=O stretching
1650 -----	1650 -----	~ 1635 1577	C=C stretching of vinyl group OH bending
1402	1400	~ 1492 1367	C-H bending C-H bending
1109 1043	1109 1043	---- ~1134	C-O-C stretching C-O-C stretching
839 769	839 769	---- 794.6	C-H rocking out of plane C-H rocking out of plane

Figure 3 show the transmission ratio Tr of the wave numbers 1787 , 2800 , 839 and 1650 Cm<sup>-1</sup> relative to wave number 792 Cm<sup>-1</sup> for each samples of EC , ER<sub>1</sub> and ER<sub>2</sub> samples of CR-39 detector . Transmission ratios Tr of the wave numbers 1787 , 2800 , 839 and 1650 Cm<sup>-1</sup> named to Tr<sub>1</sub> , Tr<sub>2</sub> , Tr<sub>3</sub> and Tr<sub>4</sub> calculated by following equations respectively .

$$*Tr_1 = \frac{[T\%]_{1787}}{[T\%]_{792}}, \quad Tr_2 = \frac{[T\%]_{2800}}{[T\%]_{792}}, \quad Tr_3 = \frac{[T\%]_{839}}{[T\%]_{792}}, \quad Tr_4 = \frac{[T\%]_{1650}}{[T\%]_{792}} \quad (1)$$

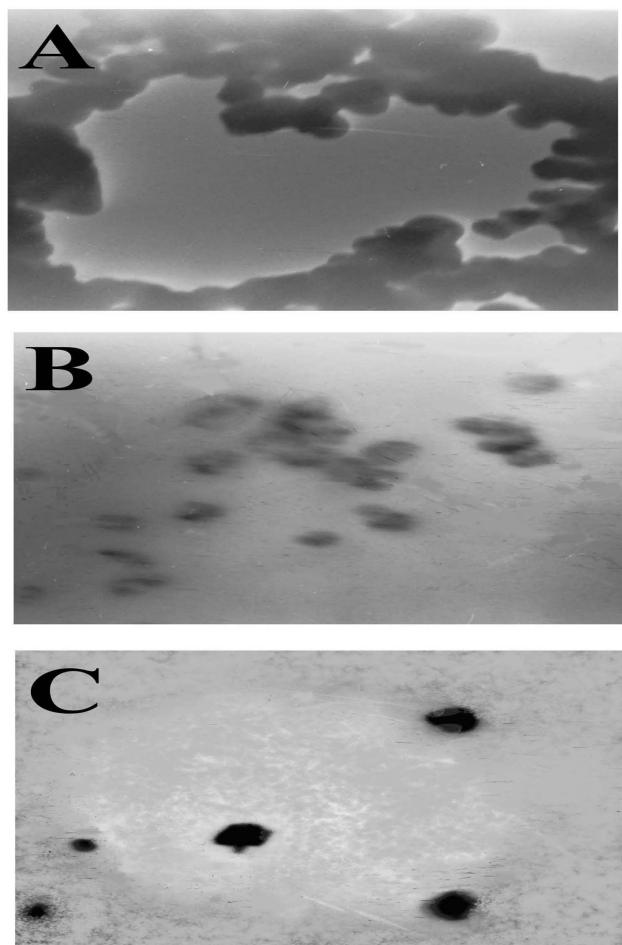
The values of Tr<sub>1</sub> , Tr<sub>2</sub> , Tr<sub>3</sub> and Tr<sub>4</sub> were dropping with increase of time of irradiation of  $\alpha$ - particles as shown in fig. 3 .



**Fig. 3 : Transmission percent ratios Tr<sub>1</sub>, Tr<sub>2</sub>, Tr<sub>3</sub>, and Tr<sub>4</sub> (1) for wave numbers 1787 , 2800 , 839 and 1650 Cm<sup>-1</sup> relative to wave number 792 Cm<sup>-1</sup> respectively . EC , ER<sub>1</sub> and ER<sub>2</sub> samples to un-irradiated , 0.3 h irradiated and 23 h irradiated for  $\alpha$ - particle irradiation of CR-39 detector respectively**

Tr<sub>1</sub> and Tr<sub>3</sub> for C=O group stretching and C-H group rocking respectively were a good agreement for radiation response better than Tr<sub>2</sub> and Tr<sub>4</sub> for CH<sub>2</sub> group starching and C=C respectively , figure 3 .

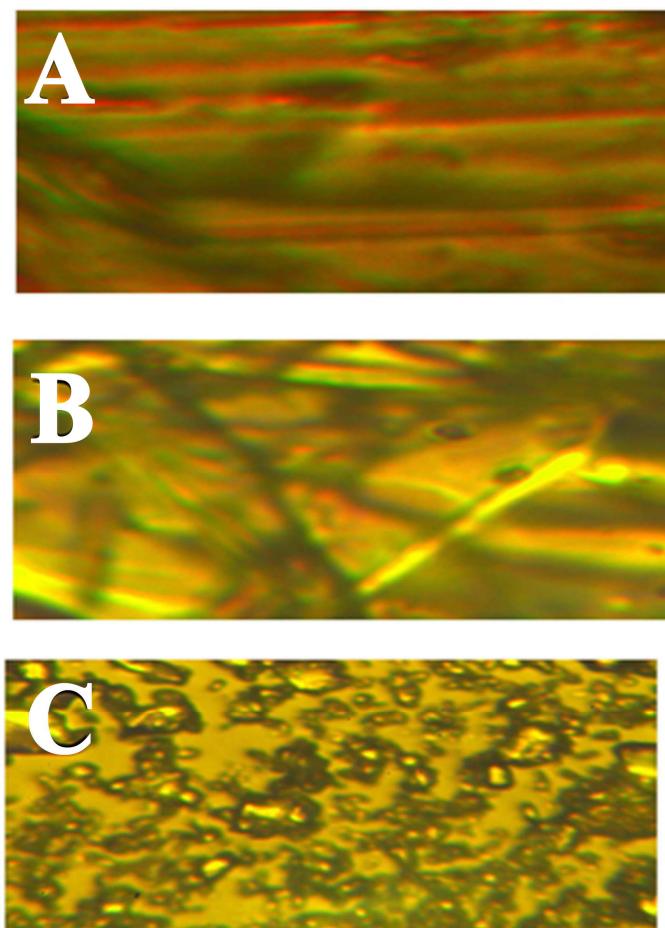
For high doses of  $\alpha$ - radiation at time of irradiation more than 23 h represent degradation for all chemical etching products to low organic compound as appearing the stability of Tr<sub>1</sub> , Tr<sub>2</sub> , Tr<sub>3</sub> and Tr<sub>4</sub> after the irradiation time 23 h . And the degradation of organic compounds with increase of irradiation time was clear appearing in the images of transmission electron microscope - TEM as shown in figure 4 .



**Fig. 4** : Transmission microscope -TEM\* images for organic etching products of EC ,ER<sub>1</sub> and ER<sub>2</sub> samples after  $\alpha$ - particle irradiated of CR-39 detector respectively . A: un-irradiated , B: 0.3 h irradiated , C: 23 h irradiated .  
 \* Transmission electron microscope -TEM model Philips CM10

The average diameter ranges of organic compounds which measured by TEM for sample B and C were 70-90 nm . The  $\alpha$ -radiation effect below irradiation time 0.3 h , figure 4 may be assessment the radiation effect for low doses by measured of relative transmission percent  $Tr_1$  and  $Tr_3$  for 1789 and  $1650\text{ Cm}^{-1}$  respectively equivalent to image analysis process by programming software for diameter ranges . The POM - analysis of inorganic compounds of chemical etching products were appear these compounds as layers at un-irradiated samples as shown in the image A for EC sample , figure 5 .

When  $\alpha$ -irradiation at time 0.3 h for ER<sub>1</sub> sample the crystalline layers were broke to small crystal as shown in the image B for ER<sub>1</sub> sample . while  $\alpha$ - irradiation at time 23 h these small crystal were degradation to vary small crystal as shown in image C for ER<sub>2</sub> . The degradation of small crystals in image C , Figure 5 also may be measured by image analysis process using programming software to produce good agreements which reflected the effect of  $\alpha$ - radiation on CR-39 detector. From this study shown the properties of chemicals etching products after  $\alpha$ -irradiation CR-39 detector with  $\alpha$ - particles for 0.3 h and 23 h was change depended on the time of radiation , and these properties measured by FTIR spectroscopy and transmission electron microscope -TEM for organic etching products were better than the analysis of inorganic crystalline etching products by Polarized microscope - PM .



**Fig. 5** : Polarized optical microscope – POM\* images for inorganic crystalline etching products of EC , ER<sub>1</sub> and ER<sub>2</sub> samples after  $\alpha$ -particle irradiated of CR-39 detector respectively . A: un-irradiated , B: 0.3 h irradiated , C: 23 h irradiated .

\*Polarized optical microscope-POM , model - Meiji MT9000 .

## CONCLUSION

The physiochemical properties which measured by FTIR spectroscopy and transmission electron microscope-TEM for organic etching products for CR-39 track detector after  $\alpha$ - particles irradiation were better than the analysis of inorganic crystalline etching products by Polarized microscope-PM .

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