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The Investigation of ESR Dating by Using Alpha-Rays

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ESR ages of the samples from Bolu Tunnel and the valley at Gerede and Gokoren from the NAF (North Anatolian Fault) are calculated by using alpha rays. The results are compared to those of the samples from the NF (Nojima Fault).

Equivalent doses (D_Es) for Al and E' centers are discussed for unetched and HF etched samples (include paramagnetic centers formed by α-, β-, γ-rays and those formed by β- and γ-rays, respectively) as $(D_E)_{\alpha+\beta+\gamma}$ and $(D_E)_{\beta+\gamma}$. Using the differences between these and dose rates of the host sediments assuming only α-rays, T values of these samples are calculated. D_Es for Al and E' centers in HF etched samples are larger than those of unetched samples except for two sets of the sample from NF. The calculated D_Es for unetched and HF etched samples are 20±4 and 4.5±0.5 kGy for Al center and 17±3 kGy and 8±2 kGy for E' center in N from NF. Calculated T_α s are as 53±23 My for Al and 30±15 My for E' by using D_α (α-dose rate) and $(D_E)_\alpha$.

Keywords
ESR dating, fault, quartz, alpha rays

1. Introduction

Dating of a geological fault formation or movements have been carried out for the risk assessment of future fault movements. Ikeya et al. (1982) have developed a method of estimating the time of fault formation or the last movement by ESR (Electron Spin Resonance) technique. ESR is useful for the study of natural radiation damage in geological materials and for dating. Dating of fault movement is based on the premise that the lattice defects produced by natural radiation in quartz, around the fault plane at the time of fault formation, are annihilated by frictional stress and temperature rise. It is assumed that the clock time was set to zero when fault formed and after that, natural radiation produces additional lattice defects. In practical dating procedure, γ-ray irradiation is performed in order to deduce the natural dose accumulated in the mineral by extrapolating the growth curve or line to the zero ordinate. The obtained dose, D_E is divided by natural dose rate, D derived from the analysis of radioactive contents in the host sediment and then ESR age, T is calculated (Ikeya, 1993).

In practice, D_E s are assumed as formed by α-, β- and γ-doses with D dose rate as the total of D_α, D_β and D_γ. α-particles have a relatively short average range of about 20μm in a medium such as quartz and form tracks along which the high ionization rate saturates all available traps. Therefore, they are less efficient than β- and γ-irradiation of the same energy in producing trapped electrons and this is expressed by the α-efficiency (Grün, 1989).

α-efficiency is determined by a calibrated monoenergetic α source. However, the determined α-efficiency does not represent the true α-effectiveness of a natural α-spectrum, since at low α-energies the rate of electron trapping is lower than at higher energies. For that reason, in some cases, HF acid is used for the reduction of α-dose from the clay matrix, during dating procedures. Therefore, α-dose effect to D_E is neglected.

In this research communication, the calculation of ESR age of a fault was attempted using only α-rays. For this, ESR ages of the sample from NAF and NF are estimated for unetched and HF etched samples. D_Es, obtained from growth curves for unetched and HF-etched samples are called as $(D_E)_{\alpha+\beta+\gamma}$ and $(D_E)_{\beta+\gamma}$. The difference, $(D_E)_\alpha$ is divided by dose rate obtained by only α-dose, $(D)_\alpha$ to calculate the age of the last movement of

Table 1: Some properties of the samples, obtained from NAF and NF, used in this study.

Samples	*Origin*	*Depth from surface (m)*	*Properties of the sample*
B1	NAF Zone	190	Meta sediment, including clay minerals and chloride, grey color, very hard.
GG	"	from the gouge at fault plane surface	light grey color, containing plenty of quartz and rare amount of plagioclase.
GOK	"	"	Limestone (partly marble), white color, containing quartz, plagioclase and calcite.
N	NF Zone	"	white clay on the surface, lime stone, white color, main component of quartz and plagioclase.

the fault.

2. Experimental:

2.1. Sample Preparation

GG, GOK and B1 core samples from Bolu Tunnel were collected from the NAF in Turkey in addition to the sample N from the NF at Hirabayashi in Japan. The properties of the samples are seen in Table 1. Samples are in bulk composition containing clay, quartz, plagioclase, chloride, lithic fragments and limestone. Quartz grains were separated from bulk composition by using several separation techniques. The samples were first immersed in water for 1 day and then washed in ultrasonic cleaner to remove clay minerals. Particles smaller than 1.5 mm were selected for further separation procedures. The samples were washed at room temperature in 10% H_2O_2 and 12 M HCl for a few hours to dissolve the carbonate matrix and then separated from the magnetic fraction using magnetic separation procedures. The samples were dry sieved to fractions of grain size. Samples of N were sieved to the fraction of <0.045, (0.045-0.075) and (0.075-0.100) mm while samples B1, GG and GOK were grouped in the size of (0.045-0.075) mm.

Each grain size of the sample was etched by using 40% hexafluorosilicic acid (H_2SiF_6) solution for 6 hours at room temperature and then rinsed with deionized water to remove remained plagioglase and its powders. The selected grains as quartz are almost pure except for a small amount of plagioclase as confirmed by X-ray diffraction analysis. These samples are further divided into two groups. First group contains α, β and γ dose effects and called as unetched samples by HF acid. The second one was additionally etched in 20% HF acid solution for 2 hours to reduce α dose from the host matrix. Thus, the second group was assumed as having only β- and γ-effects.

The weight of each grain size (≅200 mg) for all samples was decreased due to its own decreasing ratio after the treatment of HF etched. Therefore, the weights of HF etched samples were determined according to the ratios of 48, 28, 26% for GG, GOK and B1 in addition to 81, 62 and 52% for the size of <0.045, (0.045-0.075) and (0.075-0.100) mm of N sample. Aliquots of each grain size were irradiated with a ^{60}Co-source with 0.73 Gy/s of γ dose rate.

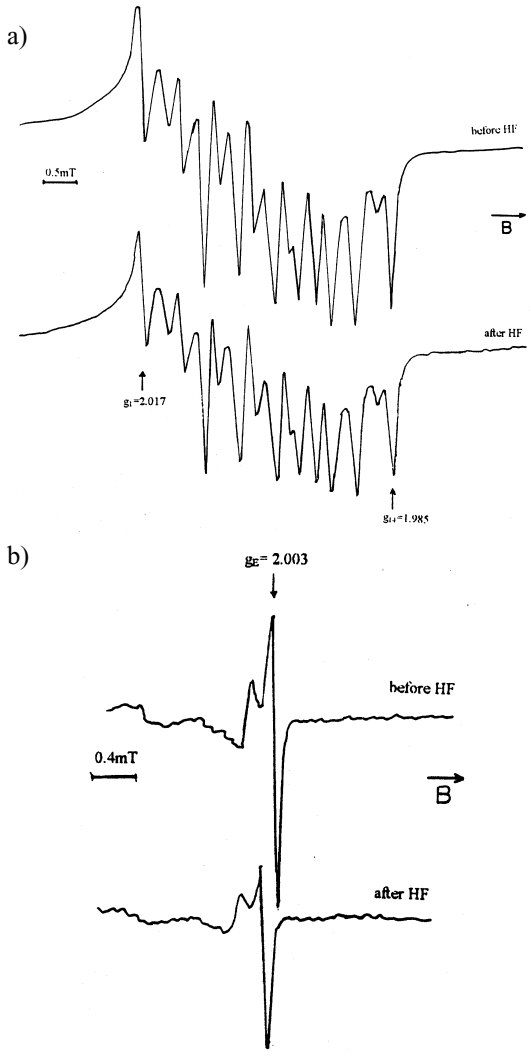

Fig. 1: ESR spectra observed in quartz extracted from the fault gouge in sample N. a) Al centers in unetched and HF etched (0.075-0.100 mm) at 77 K. b) E' centers in unetched and HF etched (0.045-0.075 mm) at room tem-

Table 2: ESR analytical data for fault gouge samples obtained from NAF and NF.

Sample	*grain size (mm)*	*D_E(kGy)* *Al*	*E'*	*Radioactive contents* *U(ppm)*	*Th(ppm)*	*K(%)*	*W(%)*	*Dose rate (mGy/a)*	*T (Ma)* *Al*	*E'*
B1				4.23±0.08	19±4	3.30±0.05	0.95			
Hexaf	0.045-0.075	3.5±0.4	8±1					6.0±0.3	0.6±0.1	1.4±0.3
HF	0.045-0.075	17±5	9±3					5.3±0.3	3±1	1.7±0.7
N				0.04±0.00	12.5±0.8	0	1.76			
Hexaf	<0.045	2.3±0.3	14±6					1.50±0.04	1.6±0.2	9±4
HF	<0.045	3±1	14±3					0.96±0.04	3±1	15±4
Hexaf	0.045-0.075	12±9	***17±3***					1.24±0.04	bad data	***14±3***
HF	0.045-0.075	15±4	***8±2***					0.94±0.04	16±5	***8±3***
Hexaf	0.075-0.100	***21±4***	16±6					1.22±0.04	***16±4***	13±5
HF	0.075-0.100	***4.5±0.5***	22±10					0.93±0.04	***4.8±0.8***	24±12
GG				2.65±0.04	15.5±0.4	0	8.14			
Hexaf	0.045-0.075	3.6±0.6	9±2					2.10±0.02	1.7±0.3	4.1±0.8
HF	0.045-0.075	bad data	9±1					1.64±0.02	bad data	5.7±0.8
GOK				1.39±0.07	2.8±0.1	0.01±0.00	1.93			
Hexaf	0.045-0.075	3.0±0.8	6±2					0.70±0.01	4±1	8±3
HF	0.045-0.075	13±7	25±1					0.54±0.01	24±13	45±3

2.2. ESR Measurements

The ESR spectra were obtained using an X-band ESR spectrometer (JEOL RE-2X) with a 100 kHz field modulation. Instrumental settings for the spectra were: microwave power = 5 and 0.01 mW; scan width = 30 and 15 mT; scan speed = 7.5 and 3.75 mT/min; modulation amplitude = 0.1 and 0.05 mT (at liquid nitrogen and room temperatures, respectively) and time constant = 0.1 second.

The Al spectra in N sample (Figure 1a) is observed only at 77 K (*g* value range 2.017 to 1.985), while E' centers are measured at room temperature, at the g values of 2.003 (Figure 1b). Therefore, ESR signal intensities of the Al and E' centers are measured to obtain the decreasing ratios of unetched and HF etched samples as about 25%. The ESR intensities are used to obtain the growth of the signals in response to the added doses. Equivalent doses (D_E) with errors were determined by extrapolating the ESR signal growth line or curve to the zero ordinate using a software. The age is calculated dividing D_E by D.

Annual doses were calculated from the contents of ^{238}U, ^{232}Th and K in the samples using γ-ray spectroscopy (Table 2). In this study, we used conversion factors assuming radioactive equilibrium in the ^{238}U and ^{232}Th series (Ogoh et al., 1993). The corrections for α- and β-attenuations for each grain size (Mejdahl, 1979 and Grün, 1989) were measured using an α-efficiency of 0.07 in quartz (Bell, 1980) and water effects were considered ignoring the contribution of cosmic rays. The contents of U, Th, K_2O, water and D with their associated errors for each sample are presented in Table 2.

3. Results and Discussions

The intensities of E' and one of the first 4 lines of Al in both unetched and HF etched quartz separated from the fault gouge increased with artificial γ-irradiation. D_E s for the samples B1, GG, GOK and N were obtained by least square fit and then extrapolation. Figure 2a shows the variation of signal intensity in sample N, having the grain size of (0.075-0.100) mm, with γ-irradiation dose for Al centers in unetched and HF etched samples, while Figure 1b is for E'centers in these samples. As shown in this figure, D_E of Al in unetched sample is larger than that of HF etched; while D_E of Al in other HF etched samples from NAF and NF are larger than those of unetched sample. In the case of E' center in N sample with the grain size of (0.045-0.075) mm, only one sample has D_E for unetched sample larger than that of HF etched (see Table 2).

These results suggest that only the samples for which α-ray reduction was observed can only be used for calculating the age of the last movement of the NF. D_α, formed by α-rays only, for Al in unetched and HF etched samples are 15±4 kGy, 0.29±0.04 mGy/y. Therefore, age of the last movement of fault, T_α, can be calculated as 53±23 My by using Al centers in N sample with the grain size of (0.075-0.100)mm. Using E' center in N with the grain size of (0.045-0.075) mm, the calculated values of $(D_E)_\alpha$, D_α and T_α are 9±3 kGy, 0.30±0.04 mGy/y and 30±15 My, respectively. However, the present values of T_αs for Al and E' are overestimated considering geological researches for this region (Ketin, 1969; Toksöz et al., 1975).

This observation may be due to the fact that the time of HF etching is short to remove the surface including paramagnetic defects formed by α-rays. So, this suggests that HF etching procedure for reduction of defects formed by α-rays should not be completed. However, Bell and Zimmerman (1978) used 40% HF for 40 minutes to remove 6 μm surface of about 100 μm diameter fraction of the quartz. It is generally assumed that after

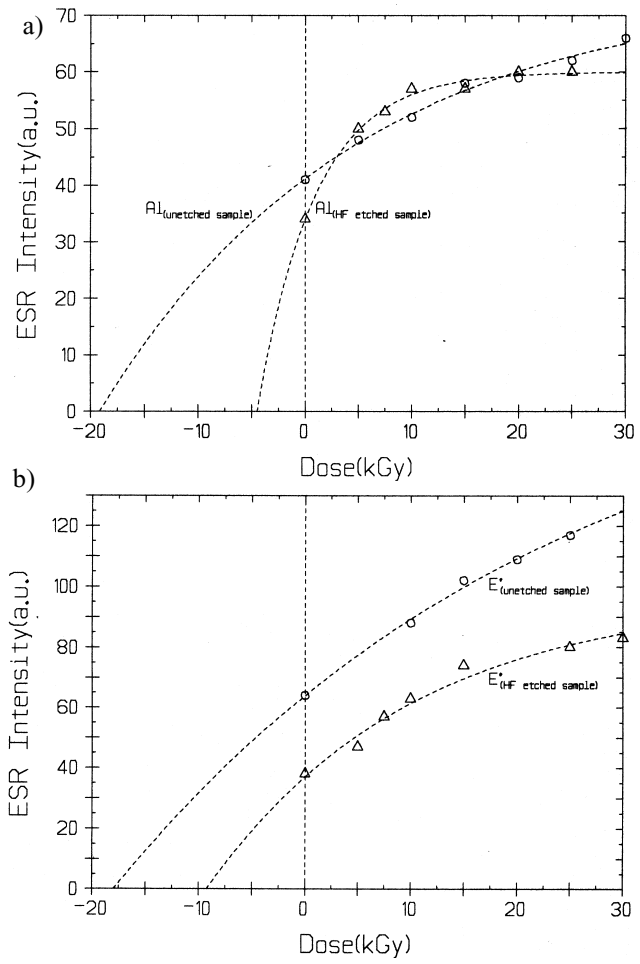

Fig. 2: ESR signal intensity vs. artificial gamma dose for unetched and HF etched samples of N. Back extrapolation through the artificially generated doses and natural dose to zero ESR intensity, define the equivalent doses $(D_E)_{\alpha+\beta+\gamma}$ and $(D_E)_{\beta+\gamma}$ for the unetched and HF etched samples. a) Al center. b) E' center.

HF treatment, the α-dose is negligible. Assuming isotropic removal, weight loss experiments on Norwegian quartz indicated that this treatment removes a surface layer of about 6 μm, corresponding to a calculated reduction in the α-dosage of about a factor of two (Bell and Zimmerman, 1978).

The other possibility for the overestimation comes from uncompleted separation procedures of quartz though the X-ray diffraction analyses done after the separation procedure indicate clearly quartz peaks. Thus, the spectra of Al and E' centers may not represent the actual growth curve and D_es.

Hence, discordant $(D_E)_\alpha$ s and T_α s of both Al and E' centers in all samples obtained from this study are not accurate. A more detailed study may be needed to calculate the date of the last fault movement.

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