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Temperature quenching mechanism of cathodoluminescence in forsterite

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Cathodoluminescence (CL), the emission of visible light caused by electron irradiation, has been widely applied to geoscience by the reason of high detection sensitivity of emission center such as impurity elements and various structural defects. Recently, CL zoning of the forsterite in carbonaceous meteorites has been investigated to clarify the origin and thermal history of the chondrules. In this case, small amount of divalent Fe ions as quencher easily eliminate CL emissions caused by impurity and defect centers, so only near end forsterite can show CL emissions. Sample temperature is one of most important effects on CL in minerals, but has not been investigated to date for the forsterite. In this study, the effect on forsterite CL has been quantitatively analyzed by assuming the Mott-Seitz model for temperature quenching of luminescence.

Forsterite crystals (Fo: 99.1) in basalt from Mogok, Myanmar and micro-grains (Fo: 99.7?99.8) in chondrules from Allende and Kaba meteorites were prepared for CL spectral measurements. CL spectroscopy was made by a SEM-CL system, which is comprised of SEM (JEOL: JSM-5410) combined with a grating monochromator (OXFORD: Mono CL2) at accelerating voltage of 15 kV and beam current of 1.0 nA in a beam scan mode. The sample temperature was controlled by flowing liquid nitrogen and using an embedded heater in a cryostage. All spectra were corrected for total instrumental response, which was determined using a calibrated standard lamp.

At room temperature, the CL spectra show broad emission bands at around 400 nm in blue region and at around 650 nm in red region, and pronounced emissions increasing to IR region with small emissions at around 720 nm. These bands can be assigned to structural defect, divalent Mn and trivalent Cr impurities, respectively. Their emission intensities increase with a decrease in sample temperature. In general, luminescence efficiency decreases with rising temperature due to an increase in non-radiative transitions. This phenomenon is called temperature quenching.

CL spectral peaks in energy unit were deconvoluted by Gaussian curve fitting to determine the emission component for each emission center. The emission peak in blue region can be separated into two components centered at 3.15 and 2.99 eV, and the peaks caused by Mn and Cr impurity centers can be fixed at 1.91 and 1.74 eV, respectively. By assuming the Mott-Seitz model, activation energy in each temperature quenching process can be calculated by Arrhenius plots using integral intensity of each component. The straight-line relationships in the plots resulted in each activation energy as follows; blue emission at 3.15 eV: 0.08?0.04 eV, blue emission at 2.99 eV: 0.10?0.05 eV, red emission at 1.91 eV: 0.01?0.005 eV, red emission at 1.74 eV: 0.01?0.02 eV. The values of activation energies for blue emissions caused by structural defects correspond to the vibration energy of Si?O stretching mode in the lattice, and the values for red emissions caused by Mn and Cr impurity centers to Mg?O vibration energy. It suggests that the temperature quenching energy might be transferred as a phonon to the specific lattice vibration.

Keywords: Forsterite, Cathodoluminescence, Temperature quenching



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Cathodoluminescence characterization of forsterite in Kaba meteorite (CV3)

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The Kaba meteorite is the most primitive unshocked CV fall (in 1857) collected nearby Kaba village, East Hungary. This meteorite is subdivided into Bali-like CV3 chondrite by the reason of a similarity to the Allende meteorite. This meteorite contains porphyritic olivine-, granular and radial pyroxene-, and barred olivine chondrules, which are surrounded by magnetite and sulfide assemblages. The study of the refractory inclusions (CAI) indicates maghemite with inverse-spinel structure and higher FeO content in inner with lighter oxygen isotope ratio, whereas the outer part consist of MgO-spinel and scattered perovskite grains. The Kaba meteorite experienced aqueous alteration and contains hydrous phyllosilicates with altered features in constituent materials.

Cathodoluminescence (CL) emissions can occasionally be detected from olivine, especially forsterite, in meteorites, whereas terrestrial one mostly gives no luminescence. CL spectroscopy and microscopy provide useful information about the existence and distribution of lattice defects and trace elements in materials with high-spatial resolution of a few micron meters. In this study, therefore, CL of the forsterite in various types of chondrules has been characterized to clarify origin of the chondules in the Kaba meteorites.

Highly forsteric olivine (Fo: 99.2?99.7) in the Kaba meteorite emits bright cathodoluminescence (CL), whereas the olivine containing fayalitic composition, even if only slight, shows no luminescence due to quenching effect of divalent Fe ions. Red luminescent forsterite is predominant, but occasionally zoned forsterite shows blue in the core and red in the rim. CL spectra of red luminescent forsterite have two broad emission bands at approximately 630 nm in red region and over 700 nm in red?IR region. The former band can be assigned to impurity center of divalent Mn ion as an activator. The latter one shows a magnificent red emission in a wide range of wavelength responsible for trivalent Cr ions, which possess two components of Cr activator and structural defect caused by interstitial Cr ions. CL spectra from blue luminescent area in the core give a characteristic broad band emission at approximately 400 nm, also associated with minor red emissions related to Mn and Cr ions. EPM analysis reveals that the concentrations of Ca, Al and Ti increase toward the center of the grain. In this case, the quenching effect of divalent Fe ions on CL might be slight and homogeneous over a forsterite grain due to low and unvaried concentration of Fe. Therefore, CL color variation observed in Kaba forsterite should be attributed mostly to intrinsic structural defect, of which distribution cannot be detected by any other methods. It implies that aqueous alteration on the forsterite might eliminate intrinsic structural defects progressively from the rim of the grain to the core, accompanied by the migration of diffusible ions of Mn, Cr and Fe to the rim where Ca and Al ions might still lie in the core. This process could proceed at low temperatures (<300?C) over a short reaction time.

Keywords: Kaba meteorite, Forsterite, Cathodoluminescence



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ESR dating of barite in sulfide deposition formed by sea bottom hydrothermal activities

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At the initial stage of studies on the seafloor hydrothermal activities, their temporal changes were not argued. However, their temporal changes, which are most probably controlled by geothermal conditions, is now an important issue in order to discuss its influence to the biosphere. For this purpose, new dating techniques are necessary, which are applied to hydrothermal minerals, in addition to conventional dating techniques.

Kasuya et al. (1991) pointed out the possibility of ESR (electron spin resonance) dating of barite (BaSO₄). *Okumura et al.* (2010) made the first practical application of this technique to the sea bottom hydrothermal barite where preliminary ages were obtained. However, in this paper, detailed discussion was not sufficient, such as the occurrence of barite included in sulfide chimney, contribution of each natural radiation from surrounding environment, optimum measurement conditions, and the stability of the signal.

In the present study, we investigated three basic issues on ESR dating of barite;(1)Optimum condition for ESR measurement (2)Thermal stability of the signal (3)Inhomogeneity of accumulated doses and dose rates.

Barite (BaSO₄) was extracted from hydrothermal chimney samples (220-E, 1354-R1, 903R7-2) taken from two sites at Okinawa Trough and at the Southern Mariana Trough. The radioactivity distribution of the chimney sample was observed in one sliced piece of chimney piece (903R7-2) to find that radioactivity is concentrated in barite with imaging plate after cutting into 13 blocks. About 2.0g of each block was crushed and was soaked in 12M hydrochloric acid. The beaker was covered with a watch glass and left for approximately 24 hours. Then, 13M nitric acid was added. Finally, after rinsing in distilled water, the sample was filtered and dried. Impurities were removed by handpicking. The extracted sample was examined by X-ray diffraction to confirm that the grains are pure barite. The dose rate was calculated assuming alpha and beta particles in an infinite system. We ignored the contribution of cosmic rays and considered the shape of the chimney about the gamma ray.

As a result, it was found that the signal of SO_3^- in barite starts to saturate at 0.01mW. Annealing experiments revealed that the signal is stable enough for age range of 10^2 - 10^3 , which are the values we obtained for present samples. The inhomogeneity of accumulated doses and of dose rates will be discussed in the presentation.

Keywords: barite, electron spin resonance, dating, hydrothermal activities



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Thermoluminescence dating of Hakusan volcanic

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Geochronological study on various events related to the magma process is important to lead to the prediction of volcanic eruptions. The Hakusan volcano, central Japan, consists of Kagamuro, Kohakusan, Shinhakusan and Uguisudaira volcanoes, and the existence of the magma chamber and related seismicity was reported to raise the alarm on the future possible eruption (Hiramatsu, 2006). The eruption age of each volcano was measured by K-Ar dating method (Higasino et al, 1984, Sakayori et al, 1999, Kitahara et al, 2006). However, some of eruptions, except for Kagamuro volcano, are relatively young within applicable age range of K-Ar dating method, so that additional dating by other dating method should be useful to reconstruct eruption history of the Hakusan volcano. This study applies thermoluminescence (TL) dating to know the eruption age of Hakusan volcano. Five samples from the Kohakusan volcano were successfully dated by the TL method. However, TL signal for andesite from Shinhakusan volcano was so small that it was difficult to be detected. The TL age for dacite from Kohakusan volcano is about 60 ka and ages from andesite are about 100 ka, concordant with the K-Ar ages reported in previous studies. The eruption activity of the Kohakusan volcano spanned more than 40 kyrs. These ages are compared to crystallization ages of zircons within magma chamber (Miyamoto et al, unpublished data) estimated by U-Th method for the samples collected from the same localities. U-Th ages are different grain by grain, though peak crystallization occurred about 30kyr earlier than the eruption age. Based on these age data, the history of Kohakusan volcano is proposed. Crystallization occurred in andesite magma and then erupted at about 95 ka. At that time, next stage of crystallization within magma chamber was going on. Somehow magma chemistry had changed from andesitic to dacitic, and then erupted at around 60 ka to bring crystals formed at the time of previous eruption.



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TL dating of marker tephras for 100 ka relevant to archeological chronology

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To determine the age of an archeological site accurately, tephra as key bed is one of the most important markers. It is effective to determine the age of tephra layer samples collected from the area close to the source vent of the tephra. The tephra sample near by the source vent, which is collected from a thick and less-altered layer, give us the most provable age of the tephra.

In this work, we try to determine the age of marker tephras using Thermoluminescence (TL) dating. We make a comparison between TL age and calibrated radiocarbon chronologies.

We think that the data set of numerical ages of marker tephras will constitute valuable contribution to the framework of the archeological chronology.

Keywords: marker tephra, TL dating, Archeological chronology, key bed



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Application of OSL dating to Tottori coastal dunes

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We examine a simplified method to obtain robust OSL age results from quartz sand samples with high recuperation based on young dune samples in the Tottori coast. Past topographic maps compiled 36 yrs and 77 yrs ago constrains depositional ages of 10 samples. A standard quartz single aliquot regenerative dose (SAR) OSL protocol was applied to these samples, but results showed age underestimation for most of them, including two that showed negative values of equivalent dose estimate. The underestimation was caused by a high recuperation due to a large contribution of the slow OSL component. To suppress the slow component contribution, we applied the early background (EBG) subtraction for estimating OSL intensity with a time window 7?19 s to a dose recovery test of 1.1 Gy for one of these samples, which successfully replicated the given dose. Thus, the EBG subtraction was applied to all of the young samples, appropriately estimating their ages except for one with a slight overestimation. Although it is uncertain what actually causes the large slow component, the method used here may be useful to analyze OSL dating results from sand samples in the Japanese archipelago that typically show high recuperation.

Keywords: optically-stimulated luminescence, chronology, Quaternary, coast, dune, sand



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OSL dating using fine quartz grains of Atotsugawa Fault

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An age evaluation of an active fault has been carried out tentatively using OSL signals emitted from crushed fine quartz grains. The tested sample was collected from Atotugawa Fault located in southern part of Toyama prefecture, which was the origin of the Hietsu earthquake in 1858.

The OSL equipment, designed by MEDC Co., Ltd and our lab, is composed of IR leaser stimulation unit (wave length:852nm) and detection unit cooperating DUG 11 filter (detection wave length:320-380nm). Different quartz grain samples in size, 63-125, 125-250, 250-355, 355-500 micron meter, were prepared for SAR dating method. Ten quartz grains were prepared for one aliquot to obtain sufficient OSL signal.

The expected equivalent dose (De) is calculated about 0.26Gy using the dose rate (1.68Gy/ka), which was estimated by U, Th, K and water content of the sample. The OSL De values lead by SAR method shows the centered value between 0.1 and 0.2Gy or value less than 0.1Gy.

Keywords: OSL dating, active fault, quartz grain, SAR method

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Luminescence dating of fine grained sediments from Lake Hovsgol, Mongolia

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Sediment core samples are recovered from the lake bottom, and meaningful proxy records to represent surrounding environment are obtained along its depth. To convert the depth profiles to the age profiles, the age model determination plays an important role. This study aims to develop the new luminescence dating technique for small amount of samples of lake sediments and the technique must supply ages easily and quickly to provide many dated horizons in a core sample. For indirect annual dose calculation, new pellet making procedures is examined, and then laser ablation - inductively coupled plasma - mass spectrometry (LA-ICP-MS) is applied to measure the concentrations of radioactive isotopes (⁸⁷Rb, ²³²Th, ²³⁴U and ²³⁸U). To investigate that the red thermoluminescence (RTL) from inorganic carbonate is capable of giving a reliable estimate of accumulated (or equivalent) dose since its deposition on the lake floor, the present work compare the result from RTL considering the residual dose of calcium carbonate from Lake Hovsgol to that estimated from conventional fine-grained quartz optical stimulated luminescence (OSL). Both equivalent doses are concordant considering the error range in most samples except for some samples which have particular reason, such as low equivalent doses and existence of authigenic carbonate. The depth profiles given based on luminescence ages are concordant with the age model of the other core sample from Lake Hovsgol and analyzed core has the environmental information from late MIS 5 or early MIS 4 to LGM.

Keywords: OSL, RTL, Lake sediments



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Thermoluminescence color image and paleoenvironment change in sediments of Lake Hovsgol, Mongolia

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The seasonal climate differences in continental interior are very intense due to the heat balance. Lake Hovsgol in Bikal Rift Zone is located on high plateau of Mongolia and has a small water catchment, which make the lake sensitive to insolation. The sediments of Lake Hovsgol record paleoclimate change in good condition because it has only one outlet at its south end, Egiin river, without influence of human activity. East Eurasia continent including this area have unique geological formation, mainly which controls the climate and environment of here, so specific information of local climate change is needed for understanding the mechanism of climate change in the entirely supracrustal.

A lot of proxies in core samples (e.g. diatoms, mineral particle size, biogenic SiO2, pollens, trace elements) have been used for reconstructing paleoclimate. In this study, thermoluminescence color image (TLCI) analysis is applied to continuous lake sediments core (HDP-04) and the validity as a new proxy is investigated.

TLCIs are recorded by digital camera and the images are converted into numerical information by using developed application. The color pixels are plotted on CIE (Commission Internationale de lEclairoge) chromaticity diagram and counted within a color zone for statistical color analyses. The diagram makes us understand the characteristic of TLCI visually.

TLCI analytical results for Lake Hovsgol sediments are ;

1) The emission intensities of TLCIs were different from sample to sample, though TLCIs showed poor reproducibility based on the large standard deviations of emission intensities on the same samples.

2) The emission intensity of TLCI and the amount of HCl-soluble-material showed similar fluctuation and the color pixel points on CIE chromaticity diagram fell in the same region with those for calcium carbonate from Darkhad basin.

3) The emission intensities of coarse samples were higher than that of fine samples.

4) A small number of color pixel points were found on blue-purple region apart from main cluster of pixels in the CIE diagrams of fine samples, however, we couldn't find the concrete evidence of presence of eolian dust fractions due to few samples analyzed.

5) The emission intensity seemed to have no relation with glaciation or insolation cycles except for several events, in which the emission intensity and the rate of red emission were decreased.

6) By spectrum analysis, the emission intensity shows cycles in connection with insolation as was found in HCl-solublematerial and grain size fluctuation.

The emission intensity does not fluctuate irregularly and must reflect amount of HCI- soluble-material and something else. More investigations of the factor having an influence on the emission intensities of TLCIs are needed. In addition, we must consider the most suitable color threshold on CIE chromaticity diagram for sample characterization. It is suggested that TLCI analysis will be valuable as a proxy of climate change in the future.

Keywords: thermoluminescence, Lake Hovsgol, paleoenvironment, HCl-soluble-material



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Temporal change of the sources of aeolian dust delivered to East Asia revealed by ESR signals in quartz

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Change of atmospheric circulation system in the past is an important issue for studies of paleoclimate. Aeolian dust, fine particle suspended in air and brought by wind, is a clue to know the wind direction and the strength in the past. Historical record of aeolian dust accumulation in Japan will give important information on this issue as well as on the climate change in the arid source regions in China. Various features, such as grain size distribution, mineral compositions, and isotope compositions, have been investigated for loess sequence for this purpose. In the present study, we would like to propose that ESR signals in quartz are other good proxies as well.

Electron spin resonance (ESR) detects unpaired electrons in minerals, trapped at lattice defects and impurities. In quartz, we observe the signals due to the E_1 ' center, an unpaired electron trapped at an oxygen vacancy, the Al hole center, an electronic hole at aluminum impurity atom replacing a silicon, the Ti centers, and the Ge center. It was shown previously that, by measuring the number of oxygen vacancies, the precursor of the E_1 ' center, in loess sequence, the aeolian dust source in Holocene was different from that in LGM (Toyoda and Naruse, 2002). Later, the crystallinity index was found to be another proxy for such studies. Temporal change of the origins of the eolian dust accumulated in the Sea of Japan was found to be correlated with the climate change by using these proxies (Nagashima et al., 2007).

The dust samples taken at meteorological observatories in Japan since 1960's were examined by the same method. It was found that finer grain fractions contain more dust component of Chinese origin than coarser fractions and that the ratio of the Chinese component varies year to year.

The number tends to decrease with time (1964-1990) with some variation from 9 to 1.5 $(1.3 \times 10^{15} \text{ spins/g})$ in June and from 3 to 1.8 in March. The number in June is higher than the one in March in the 1960s while it is opposite in the 1970s. The decrease of the number of oxygen vacancies can be due to change in sources of dust in China.



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Standard procedures for evaluing the oxygen vacancies and the crystallinity index in quartz: applications to provenance

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The number of oxygen vacancies, obtained by ESR (electron spin resonance) measurements, and the crystallinity index, obtained by X ray diffraction studies, have been found to be good proxies in estimating the origin of aeolian dust. The variations of origins of aeolian dust were investigated using these techniques to discuss the changes related to temporal changes in climate and monsoon by examining the loess sequence in Japan and the sediments in Japan Sea.

However, the methodology of these techniques has not yet been established. For example, gamma ray dose which is thought to be necessary to evaluate the number of oxgen vacancies by ESR, and scan speed and scan range vary depending on the researchers. In case of fine atomospheric deposition, it is very difficult to obtain pure quartz by chemical treatment. Therefore, the value is obtained by dividing the observed value by the quartz content, which is measured by X ray diffraction. It may be an issue to examine to test if the value varies depending on the quartz content. In the present study, we study the best measurement parameters for these techniques.

Keywords: ESR, crystallinity index, quartz, provenance