



CATHODOLUMINESCENCE-BASED LABORATORY ASTROPHYSICS

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CPS, KOBE, JAPAN 23rd JANUARY 2013

PURPOSE

Cathodoluminescence and its implication for the Geosciences and Material Sciences have already been described by previous studies, but the application to the Laboratory Astrophysics has not been debated in details, up to date.

This overview talk is to provide a summary of the preliminary examinations of the cathodoluminescence investigations (as potentials of this technique) in the laboratory analogous materials providing some important information about the possible formation mechanism of forsterite in the Early Solar System and diamonds in the planetary nebula as well as determination of shock wave history of the fine-grained astromaterials, respectively.

Following a systematic scheme as follows:

Astronomical observation- Scientific question-Cathodoluminescence investigation of the experimentally-grown samples and natural specimens-Conclusions



Introduction to Cathodoluminescence

Experimental Procedure

Cathodoluminescence microcharacterization of forsterite in nature and experiment and its application to meteoritics and astromineralogy

CL properties of micro-and nanodiamonds and their implication for astrophysics

Shock wave history of the fine-grained materials

BASICS OF CATHODOLUMINESCENCE (based on a persoal communication with Professor Jens Götze, at University of Freiberg, Germany) **Basics of luminescence**

Luminescence

= transformation of diverse kinds of energy into visible light

Luminescence of inorganic and organic substances results from an emission transition of anions, molecules or a crystal from an excited electronic state to a ground state with lesser energy.

(Marfunin1979)

Main processes of luminescence

- (1) absorption of excitation energy and stimulation of the system into an excited state
- (2) transformation and transfer of the excitation energy
- (3) emission of light and relaxation of the system into an unexcited condition

Basics of luminescence

Primary electron beam



Basics of luminescence



Electron beam interaction with a solid



Götze (2000)

Basics of luminescence

The band model



Basics of luminescence

Visualization of the "real" structure of solids by CL





intrinsic

lattice defects (broken bonds, vacancies) extrinsic

trace elements (Mn²⁺, REE^{2+/3+}, etc.)

Types of luminescence centres

- transition metal ions (e.g., Mn²⁺, Cr³⁺, Fe³⁺)
- rare earth elements (REE^{2+/3+})
- actinides (especially uranyl UO₂²⁺)
- heavy metals (e.g., Pb²⁺, Tl⁺)
 - electron-hole centres (e.g., S₂⁻, O₂⁻, F-centres)



Mineral groups and minerals showing CL

in general all insulators and semiconductors

elements	diamond
sulfides	sphalerite
oxides	corundum, cassiterite, periclase
halides	fluorite, halite
sulfates	anhydrite, alunite
phosphates	apatite
carbonates	calcite, aragonite, dolomite, magnesite
silicates	feldspar, quartz, zircon, kaolinite

- technical products (synthetic minerals, ceramics, glasses !)
- no luminescence of conductors, iron minerals and Fe-rich phases

Experimental Procedure

Luminoscope (ELM-3R- by excitation voltage at 10 kV and beam current of 0.5 mA at vacuum condition under 100 mTorr.) with a CCD camera.

SEM (JEOL: JSM-5410LV) combined with a grating monochromator (OXFORD: Mono CL2), which has the following characteristics: 1200 grooves/mm, a focal length of 0.3 m, F of 4.2, limit of resolution of 0.5 nm, and slit width of 4 mm at the inlet and outlet.

The dispersed CL was recorded by a photon counting method using a photomultiplier tube (Hamamatsu: R2228) and converted to digital data.

All CL spectra were corrected for total instrumental response using a calibrated standard lamp (Eppley Laboratory: Quartz Halogen Lamp).

The corrected CL spectra in energy unit were deconvoluted into the Gaussian component corresponding to each emission center using a peak-fitting software (Peak Analyzer) in OriginPro 8J SR2.





CATHODOLUMINESCENCE MICROCHARACTERIZATION OF FORSTERITE IN NATURE AND EXPERIMENT AND ITS APPLICATION TO METEORITICS AND ASTROMINERALOGY

According to Bouwman et al. (2008) ApJ 683 479

"we find a change in the relative abundance of the different crystalline species: more enstatite than forsterite is observed in the inner warm dust population at ~1 AU, while forsterite dominates in the colder outer regions at ~5-15 AU. This change in the relative abundances argues for a localized crystallization process rather than a radial mixing scenario in which crystalline silicates are being transported outwards from a single formation region in the hot inner parts of the disk."

What can we learn from the super cooling crystallization of forsterite?

Can we find forsterite formed at super cooling conditions in the primitive meteorites?

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Journal of luminescence 132 (2012) 1011-1047

Contents lists available at SciVerse ScienceDirect

Journal of Luminescence

journal homepage: www.elsevier.com/locate/jlumin

Cathodoluminescence microcharacterization of forsterite in the chondrule experimentally grown under super cooling

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ABSTRACT

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Antide Narozy: Rocei und 2 October 2011 Rocei und in revised form 26 November 2011 Accepted 1 December 2011 Available online 9 December 2011

Repeards: Supercoding Ronzenite Catholid uninescence Solar system Cathodoluminescence (CL) of laboratory is naterite chondrains has been characterized to darify the formation process of chondrains and related machanism of the crystal growth in a supersolution of the formation of the insertion structure of laboratory, which reflects to the anisotropy of crystalinatio branches of the insertion structure of laboratory, which reflects to the anisotropy of crystallization. A new CL band centered at 450–555 nm (2.76-2.26 eV) in these to given negleon might be analged to a microdelect-related center, which is a diagnostic peak for the formerise that was formed due to the radio growth as high as ~10 mm/s or higher from a supercooled mels.

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1. Introduction

According to Weisberg et al. [1] barred-olivine (BO) chordrule has a unique solidification texture containing single or multiple groupings of elongated parallel okvine crystals (barred texture) with shell of olivine (rim). Cryst al growth experiments have been tried to reproduce the barred-olivine texture, but its formation mechansim has been still not understood well [2,3]. Tsukamoto et al. [4,5] produced a rim structure in very rapid cooling experiments with forsterite melt droplets levitated aeroacustically. They observed a sudden temperature increase of hundreds kelvin during solidification of the droplet. This is due to a release of latent heat of crystallization and termed as recalescence, which played a key role for the formation of solidification textures of chondrules. More recently, numerical simulation of crystal growth in a supercooled melt droplet using pure forsterite has revealed that the rim detected by an optical examination was formed when the droplet cooled very rapidly [6]. The fast cooling of the droplet makes an accelarated chilled margin on its surface where a crystal growth can make progress sapidly resulting in the formation of the characteristic rim along the grain surface. Furthermore, the recalescence at the dropfet surface generates the "reversed" temperature gradient at crystal-liquid interface

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0022-2310/S- see front matter © 2011 Blassier B.V. All rights reserved, doi:10.1016/j.Junin.2011.12.011

sofficient to cause dendritic growth inside the droplet by morphologic insubility [6]. These observational and theoretical results suggested that the barred texture with the rim was formed by a rapid crystal growth in a second.

Cathodoluminescence (G.) spectral analysis provides valuable information on a combination of various activators and defect centers in forstentie [7–13]. CL imaging is also applied as a powerful tool to characterize or identify fostentie from different planetary materials such as meteorites, micrometeorites and Lunar rocks [14–18] as well as from the experiments [14,19].

For the synthetic and metooditic fosterike samples, however, the defect-related centers produced in various conditions during cooling and/or reasiscence have been little investigated so far. Moreover, the crystal growth condition of forsterite as one of the first crystalline particles occurred in the Early Solar System is still controversial. In this study, G. imaging and spectroscopy have been performed to characterize luminescent centers of the fosterike in the chordrafe experimentally grown under the super cooling and u discuss formation mechanism of the meteoritic fosterike.

2. Sample and experimental procedures

Forsterite crystals with 1-2 mm in diameter were grown as a chondrule analog by the aero-acoustic levitation floating method as shown in Fig. 1A [4,5]. Levitated melt crystallizes via homogeneous nucleation under very high super cooling, because heterogeneous nucleation is suppressed due to ontainer free

Based on.....





Schematic of the aero-acoustic levitator and optical path of CO2 laser for heating. A laboratory chondrule, which has been achieved by aero-acoustic levitation, crystallizes homogeneously due to container free after reducing or cutting off the power of CO2 laser.

Temperature histories, which was measured by pyrometer, during the formation of laboratory chondrule FS004 (black) and FS005 (grey). Crystallization occurs under super cooling of T1 and T2 for FS004 and FS005, respectively. Temperature history of FS013 is almost similar to FS005. Temperature elevates immediately due to latent heat by crystallization. During crystallization, temperature was maintained around a melting point, which is at 1890C for pure forsterite, for the duration of t1 and t2, respectively. TABLE 1 Cooling and growth rates as well as CL characteristics of the experimentally grown forsterite samples used in this study.

Sample	Growth Rate (µm/s)	Cooling Rate (K/s)	ΔT ('C)*	CL spectra (nm)	CL color/imaging
FS004	20-100	20-50	20-50	394, 643, 724	red/star-type features
FS005	1000	20-50	400	396,460,628	blue/dendritic texture
FS008	1000	20-50	400	395,461,628	blue/dendritic texture
FS010	1000	20-50	400	397,461,633	blue/dendritic texture
FS013	>1000	20-50	>400	409,481	green/dendritic texture
FS014	>1000	20-50	>400	401,480,638	green/dendritic texture

 ΔT value corresponds to the temperature difference between the equillibrium temperature (T_m =1890 °C) and the temperature of nucleation

Optical Microscope and CL Observations of the Red CL Forsterite Lab Chondrule



PPL



XPL



CL



Scale : 1 mm



Corrected CL spectrum (2.0 nA, 15 kV)

Corrected CL spectra



Corrected CL spectrum (2.0 nA, 15 kV)

Optical Microscope and CL Observations of the Blue CL Forsterite Lab Chondrule



PPL



XPL



CL





Corrected CL spectrum (2.0 nA, 15 kV)

Corrected CL spectra



Corrected CL spectrum (2.0 nA, 15 kV)

Optical Microscope and CL Observations of the Green CL Forsterite Lab Chondrule



PPL













Intensity vs Emission Energy







-1.75 eV Cr -1.92 eV Mn -3,00 and 3,15 eV defect center (O vacancy) -2.6-2,7 eV microdefect???



Meteoritic Forsterite

OCL image of the Tagish Lake primitive chondrite

An-going research

Conclusion: we suggest that forsterite in the cold outer regions of th protoplanetary disk was formed under the super cooling conditions



In this study, cathodoluminescence (CL) microscopy and spectroscopy of the forsterite chondrules have been characterized to understand more about the mechanism of the crystal growth under the rapid cooling condition.

The color CL image of experimentally grown forsterite exhibits significant blue luminescence in the main branches of the interior structure of lab-chondrule, which reflects an order of crystallization.

CL spectra from the blue luminescent area give a characteristic broad band emission at around 450 nm, which is associated with a relatively small concentration of Al, Ca, Ti refractory elements.

A new CL band centered at 480 nm (blue/green CL color) might be assigned to a microdefect-related center, which is a diagnostic peak for the forsterite that was formed due to the rapid growth from super cooled melt.

CL INVESTIGATION OF NANO- AND MICRODIAMONDS



Spectral Feature (Å)	Spectral Feature (nm)	Temperature (K)
4263	426,3	83
4638	463,8	83
5030	503	77
5773	577,3	77
5780	578	77

Spectral properties of the NGC 7027 planetary nebula

Origin of diamond is still poorly understood

Simonia and Mikhail, 2006

Microscopy... Microanalysis

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Cathodoluminescence Microscopy and Spectroscopy of Micro- and Nanodiamonds: An Implication for Laboratory Astrophysics

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Based on...

Abstract: Color centers in selected micro- and nanodiamond samples were investigated by cathodoluminescence (CL) microscopy and spectroscopy at 298 K [room temperature (RT]] and 77 K [liquid-nitrogen temperkure (LNT)] to assess the value of the technique for astrophysics. Nanodiamonds from meteorites were compared with synthetic diamonds made with different processes involving distinct synthesis mechanisms (chemical vapor deposition, static high pressure high temperature, detonation). A CL emission peak centered at around 540 nm at 77 K was observed in almost all of the selected clamond samples and is assigned to the dislocation defect with nitrogen atoms. Additional peaks were identified at 387 and 452 nm, which are related to the vacancy defect. In general, peak intensity at LNT at the samples was increased in comparison to KT. The results indicate a clear temperature—dependence of the spectroscopic properties of diamond. This suggests the method is a useful tool in laboratory astrophysics.

Key words: cathodoluminescence, scanning electron microscopy, nanodiamond, astrophysics

INTRODUCTION

Diamonds play an important role not only in the material sciences but also in Earth and Planetary Science. For example, nanodiamonds may be one of the most important types of stardust in primitive meteorites, with their origin linked to supernova explosions, as indicated by the isotopic compositions of trace elements that they carry (Ott, 2003, 2009 and references therein). Such nanodiamonds are also among the samples studied in the work reported here.

Color centers in diamond have been studied over the past years (e.g., Davies & Harner, 1976; Zaltsev, 2001). According to Threi and Koclak (2012), these centers are punctual defects, created by the presence of substitutional or interstitial atoms or vacancies in different configurations. Cathodoluminescence (CL) has emerged as an alternative to other methods to study individual emission centers with a better spatial resolution. To date, there are limited CL investigations of natural micro- and nanodiamonds (Grund & Bischoff, 1999; Heiderhoff et al., 2001; Kumar et al., 2006; Karzemska, 2010; Pratest et al., 2003; Orlanducci et al., 2006; Karzemska, 2010; Sopylova et al., 2010; Shiryaev et al.,

Received Merch 15, 2012; accepted August 14, 2012

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2011). The technique has been applied more frequently to synthetic diamond, for example, chemical vapor deposition (Kawarada et al., 1988; Robins et al., 1988; Yacobi et al., 1991; Katsumata, 1992; Won et al., 1996; Kanda et al., 2003), high pressure high temperature (HPHT; Katsumata, 1992; Kanda & Watarabe, 2004; Stevens-Kalceff et al., 2008), and ultradisperse detonation diamonds (Guesik et al., 2009).

Groups of diamonds should be divided into four types, which are based on the local configuration of impurities within the carbon lattice, as follows (Walker, 1979; Zaitsev, 2001). The most common diamond type is Type I in which the nitrogen concentration is around 0.1%. According to their optical absorption and luminescence properties, the Type I class is further classified into the Type Ia and Type Ib diamonds. Almost all natural diamonds belong to Type Ia, where the nitrogen impurities are dustered within the crystal lattice of carbon atoms. Such diamonds absorb blue light that provides a significant narrow band centered at around 415 nm, which is assigned to the N3 centers (where the diamonds contain clusters of three nitrogen atoms). Additionally, a relatively weak line at 478 nm is related to the N2 nitrogen center (blue fluorescence). The green band at 504 nm (H3 center) is often accompanied by a weaker shoulder or broad band centered at 537 and 495 nm (H4



Silicon colloidal polish to get a smooth surface and to avoid any carbon-related contanimation.

K2 nanodiamonds mounted in nonradiative twocomponent epoxy



SEM-CL







HPHT Natural Diamond

CVD Diamond

Synthetic Diamond D∼0.25µm



Cathodoluminescence spectra of the natural diamond at room temperature (RT) and liquid nitrogen (LNT) temperature showing a significant CL band centered at 541 nm.



CL spectrum of the synthetic HPHT-diamond sample obtained at RT exhibits a broad band at 540 nm with two shoulder peaks 448 and 729 nm (a). There is a peak shift as well as peak broadening of a band at 590 nm and peaks at 444 and 729 nm show increasing peak intensity in the LNT-CL spectrum (b).



Based on a personal communication with Prof Em Adolf Witt at the University of Toledo

ERE in the NGC 7023 might be related to the nanodiamonds.



The CVD diamond sample contains almost no CL peaks in RT (a) whereas a broad band centered at 509 nm is dominant in the LNT-CL spectrum.





Boriskino meteoritic nanodiamond shows a significant broad peak at 540 nm in the RT-CL spectrum, but its LNT-CL does not contain any peaks.

Preliminary Results

Micro-and nanodiamond samples from different origin such as Chemical Vapor Deposition, High-Pressure High-Temperature, Ultradispersive Detonation Diamonds as well as a sample of meteoritic nanodiamonds were investigated by cathodoluminescence microscopy and spectroscopy at room temperature (RT) and liquid nitrogen temperature (LNT).

A cathodoluminescence emission peak centered at around 540 nm at liquid nitrogen temperature was observed in almost all of the selected diamond samples and is assigned to the dislocation defect with nitrogen atoms.

Additional peaks were identified at 387 and 452 nm, which are related to the vacancy defect. The results indicate a clear temperature - dependence of the spectroscopic properties of diamond.

Conclusions

•diamond particles in nebula NGC7027 may be originated from the dust materials supplied by an ejection of the outer parts of the Red Giants during planetary nebula formation
•larger particles than 7 nm
•N-enriched diamonds

FUTURE WORK: CATHODOLUMINESCENCE-BASED SHOCK STAGE DETERMINATION OF THE FINE-GRAINED ASTROMATERIALS

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SHOCK METAMORPHISM

Materials subjected to shockwaves display characteristic and irreversible physical and chemical changes on both macroscopic and microscopic scales depending on the applied shock strength.

One of the most important parameters that needs to be clarified in the formation process of planets, comets and asteroids is the peak shock pressure due to the impact events.

Asteroids and meteorites that have experienced shock impacts provide valuable information on collision and accumulation of asteroid and planetesimal during planetary accretion, formation of impact crater on planet, the satellite and asteroid, and ejection of asteroid and meteorite from the parent body.



In a pioneering study, Sippel and Spencer (1970) observed that the shock metamorphism caused *peak shifts* from green peak toward the red peak, *peak broadening* and *decrease of luminescence intensity* than in the undamaged counterpart in the CL spectra of shock-metamorphosed lunar feldspars. They noted that the distortions or disorder in the crystal field results in crystal field perturbations and these local variations occur broadened distribution of excited state energies due to shock metamorphism.



CL spectral measurements were performed on natural and experimentally shocked oligoclases (An19.7 single crystal shocked between 10.5 GPa and 45 GPa) and plagioclases from the equilibrated ordinary chondrites (Dar al Gani, Tenham) (Kaus and Bischoff, 2000).

PREVIOUS STUDIES: CATHODOLUMINESCENCE, ELECTRON MICROSCOPY, IR AND RAMAN SPECTROSCOPY OF SHOCK-METAMORPHOSED ZIRCON

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Shock barometer using cathodoluminescence of alkali feldspar

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Received 14 November 2011; revised 30 July 2012; accepted 2 August 2012; published 18 September 2012.

[1] Color cathodoluminescence (CL) images of unshocked and experimentally shocked sanidine at pressures up to 40.1 GPa showed red-violet emission below 20.0 GPa and blue emission above 20.0 GPa. The phases in these shock-recovered samples were identified as crystalline feldspar for red-violet emitting areas and as diaplectic feldspar glass for blue emitting ones by micro-Raman spectroscopy. CL spectra of these shocked sanidine have emissions at ~330, ~380 and 400-420 nm of which intensities increase with an increase in shock pressure. Similar UV-blue emissions were found in alkali feldspar and the glass in Martian meteorites and Ries crater impactite. The deconvolution of these CL spectra provides the emission component at 2.948 eV assigned to shock-induced defect center, where this intensity correlates linearly with peak shock-induced pressure on sanidine, with little dependence on composition and structure. The correlation gives quantitative values of the shock pressures experienced by the feldspar, resulting in estimated shock pressures of Martian meteorites and Ries crater impactite. The CL intensity of feldspar has a potential for a universal shock barometer with high spatial resolution ($\sim 1 \mu m$) and in a wide pressure range (theoretically ~4.5-40.1 GPa). This leads to a breakthrough in understanding the impact histories on Earth, Moon, and Mars.

Citation: Kayama, M., H. Nishido, T. Sekine, T. Nakazato, A. Gucsik, and K. Ninagawa (2012), Shock barometer using cathodoluminescence of alkali feldspar, J. Geophys. Res., 117, E09004, doi:10.1029/2011JE004025.

E09004

1. Introduction

Based on.....

[2] Meteorite and impactite that have experienced impacts provide vital information on collision and accumulation processes of asteroid and planetesimal during planetary accretion, formation process of impact crater on planets and their satellites, and ejection process from the parent body [e.g., Stöffler et al., 1991; French, 2004; Beck et al., 2005; Ohtani et al., 2010]. Materials subjected to shockwaves display characteristic and irreversible structural changes on both macroscopic and microscopic scales, depending on the applied shock strength. The shock pressure is one of the most important parameters that need to be clarified in the collisional history of asteroid, meteorite and planetesimal

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impacts [e.g., Stöffler et al., 1991; Fritz et al., 2005a; Gillet et al., 2007; Kubo et al., 2010]. Various techniques such as refractive index, X-ray diffraction, infrared (IR) absorption and micro-Raman spectral analyses, as well as optical microscopic observations, have been applied to evaluate the shock pressure in minerals, predominately feldspar, which is one of major rock-forming minerals on the surfaces of Earth, Moon, and Mars. The shock pressures on meteorites and impactites have been gualitatively estimated based on the presence of characteristic features, structures or phase and on the paragenetic assembly of highpressure phases [e.g., Stöffler et al., 1986; Ostertag et al., 1986; Beck et al., 2005; Fernandes et al., 2009; El Goresy et al., 2010a; Ohtani et al., 2010]. Although X-ray diffraction analysis and IR spectroscopy of shocked feldspar have been conducted for usage as a shock barometer, they are not enough to estimate shock pressure on feldspar because the change of their features depends on many factors such as the shock-induced pressure, phase composition, the degree of Si-Al order, and grain size [Hass et al., 1978]. Raman spectroscopy combined with optical and scanning electron microscopes has been used to identify micrometer-order feldspar and maskelynite (an amorphous product changed from shocked feldspar) and to deduce briefly the degree of shock strength in Martian meteorites and impact crater [Fritz et al., 2005a]. The refractive index measurement also gives quantitatively estimated shock pressure on a few hundred micron-order feldspar grains in meteorite and impactite in the range from ~15 to 45 GPa [Lambert, 1981;

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A POSSIBLE FORMATION SCENARIO OF ITOKAWA



Based on the Prelimanary Examination published in Science (August 2011)



Cathodoluminescence and Raman spectra of the unshocked and experimentally shocked sanidine.

PRELIMINARY RESULTS

Samples	This study (CL intensity)	Previous studies
NWA 2975	34.4 ± 2.0 GPa	
Shergotty	31.3 ± 1.7	26-32 (av. 28.4)
		29 ± 1
		30.5 ± 2.5
)hofar 019	26.1 ± 1.2	26-29
		27
Zagami	25.5 ± 1.1	27
		31 ± 2
		29.3
		28-30 (av. 29.2)
		22.5
		23
		29.5 ± 0.5
Yamato 000749	7.4 ± 0.8	5-14
		5-20
Ries crater	15.9 ± 0.7	< 22
		20-22 to 28-34
		28-34 to 42-45
		~15-17

I am thankful to the collaborators of these projects as follows: Prof Akira Tsuchiyama (Kyoto University), Prof. Yuki Kimura (Tohoku University, Sendai), Prof. Katsuo Tsukamoto (Tohoku University, Sendai), Prof. Hitoshi Miura (Tohoku University, Sendai), Irakli Simonia (Georgia) and Dr Jean-Paul Boudou (Orlay, France).

I am also thankful for Sasha (Open University, UK) and Conny (Max Planck Institute, Jena, Germany for providing me the nano-and microdiamonds samples.

I am grateful to Prof Hirotsugu Nishido, Prof. Kiyotaka Ninagawa, Dr Masahiro Kayama, Mr Endo and Mr Nakazato at Okayama University of Science as well as University of Hiroshima and their tremendeous effort on the CL measurements of the selected specimens.

I am appreciated Dr Ulrich Ott's supervision and usfeful comments on my scientific activity at Max Planck Institute for Chemistry, Mainz, Germany.



Cathodoluminscence and its Application in the Planetary Sciences

MICRO-RAMAN SPECTROSCOPY AND LUMINESCENCE STUDIES IN THE EARTH AND PLANETARY SCIENCES

Proceedings of the International Conference Mainz, Germany 2-4 April 2009

> EDITOR Arnold Gucsik



AIP CONFERENCE PROCEEDINGS 1163



Thank you very much for your attention