Thermoluminescence study of Natural Dolomite Mineral

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Abstract

The town Chhotaudepur is 105km far away in the east of Vadodara, is the main source of natural mineral Dolomite, which is nothing but double carbonate of calcium and magnesium. It has a wide application area for e.g. in ceramics, glass industries, steel refractories, as filler material in the paint, in making milk of magnesia, as fertilizer to reduce the acidity of the soil and much more.The natural Dolomite sample was collected from the mines of Chhotaudepur and taken for the Thermoluminescence study. TL was recorded for as received sample and ATL was also recorded for the sample annealed and quenched from different temperatures followed by beta radiation dose, for better understanding the sample is taken for XRD, FT-IR, SEM and particle size analysis.

Keywords: Dolomite, Thermoluminescence, ATL.

1. Introduction

The town Chhotaudepur is famous for the dolomite mining and dolomite factories. The average mineral processing is around 3000 tons per day which have commercial value around 30 lacs. About 20000 people are employed in this mining activity, factories, trading and transportation and hence it is the backbone of the town's economy. The mineral dolomite is a sedimentary rock of double carbonate of calcium and magnesium which is deposited in several feet thick massive beds on the bank of river 'Orsang'. It uses in the paint as filler material, in ceramic industries as base mineral, in glass industries, in fertilizer to reduce the soil acidity and as a source of magnesium, in plastic industries to save the production cost, refractory bricks, paver blocks etc [1].

TL technique has a variety of applications such as in the detection of impurity and defect, Geological dating, personal and accidental dosimetry, growth in sedimentation activity etc [2].

The present paper reports the TL characteristics of as received dolomite mineral as well as annealed and quenched from 200, 400, 600 and 800 °C followed by irradiation of 25 Gy beta dose using Sr-90.

2. Experimental

The natural sample of dolomite is collected immediately after blasting of rocks and packed in the black bag. This was ground with agate mortar and pastel. Natural thermoluminescence is recorded for as received sample and then irradiated by a beta dose of 25Gy using Sr-90, also TL recorded for the sample annealed and quenched from 200°C, 400°C, 600°C and 800°C followed by the 25Gy beta dose irradiation. For the annealing and quenching microcontroller based furnace is used and Microcontroller base Nucleonix TL reader is used to record TL. XRD powder pattern was recorded using Brukker D2 Phaser with Cu Kα (1.54056Å) line, Thermo Nicolet 6700 was employed for recording FT-IR spectra using KBr pellet method, particle size analysis was performed using Mastersizer micro, SEM photographs were taken using Leo S-440i (EDX, Model-7060), TGA curve was recorded using Mettler Toledo

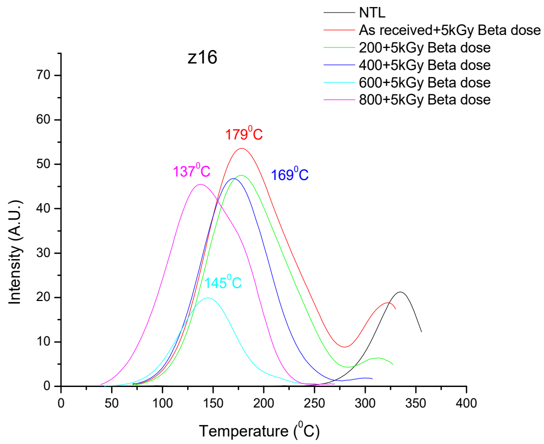
3. Results and Discussions

The particle size of the powder sample of natural dolomite was measured 45µm.

3.1 NTL and ATL of Natural Dolomite

The TL was recorded of this sample having 5mg weighed. Fig:1 shows the NTL and ATL of the as received sample annealed and quenched from 200, 400, 600 and 800 °C followed by beta dose irradiation of 25Gy using Sr-90.

Fig.1: TL of Natural Dolomite.

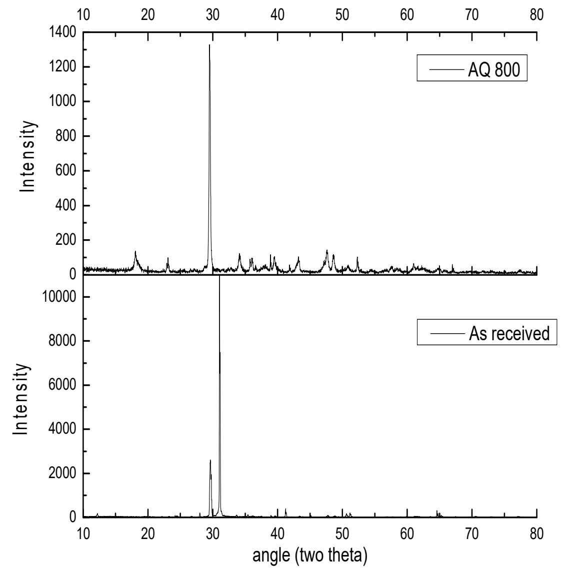


There is no NTL observed within the temperature range whereas ATL and annealed and quenched from 200°Cshows a well resolved peak around 179°C. A small hump is observed around 319°C. The variation in entire glow curve is negligible for entire glow curve. From the TL glow curve, it is clear that the TL peak shifts towards lower temperature as the annealing and quenching temperature increases from 200 to 800°C. There is a hump observed around 345°C. The intensity of this hump is decreased as the annealing and quenching temperature increases and shifts towards the lower temperature side and vanishes at the sample annealed and quenched from 800°C.

3.2 XRD of Natural Dolomite and AQ 800°C

XRD analysis shows that dolomite is the dominant mineral in the sample. The room temperature XRD pattern of sample displays sharp diffractions that attribute to dolomite (JCPDS files card 79-1342; 2000). From the XRD pattern of the as received sample, high intensity peak is observed at 31^0 and low intensity peaks are observed around 12^0, 28^0, 29^0, 35^0, 41^0, 45^0, 51^0 and 65^0. The crystallite size is calculated from Scherrer's formula and is found to be 71.80 nm.

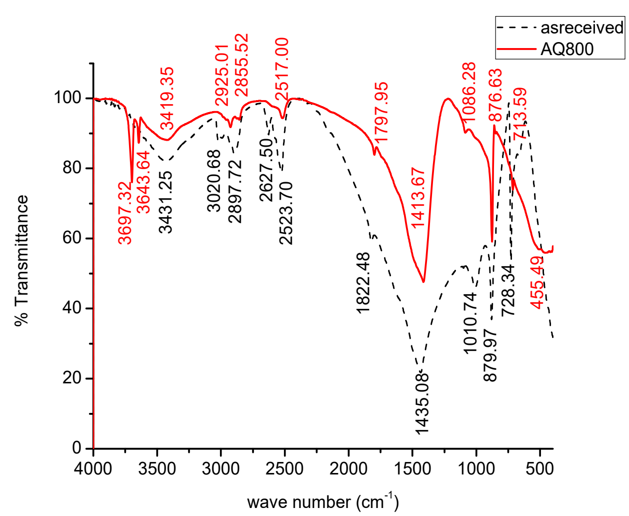
Fig.2: XRD of natural dolomite and AQ 800°C.



XRD for the sample annealed and quenched from 800^0C, high intensity peak is observed at 29^0 and low intensity peaks are observed around 18^0, 23^0, 34^0, 36^0, 39^0, 43^0, 47^0, 48^0, 52^0 and 61^0. The crystallite size is calculated from Scherrer's formula and is found to be 39.02nm, which is about 45% less than as received sample. This may be due to the release of trapped gas molecules, structural water and deformation under the partial collapse of crystal when annealed and quenched from higher temperature.

3.3 FTIR of Natural dolomite and AQ 800°C

Fig.3: FT-IR of Dolomite.

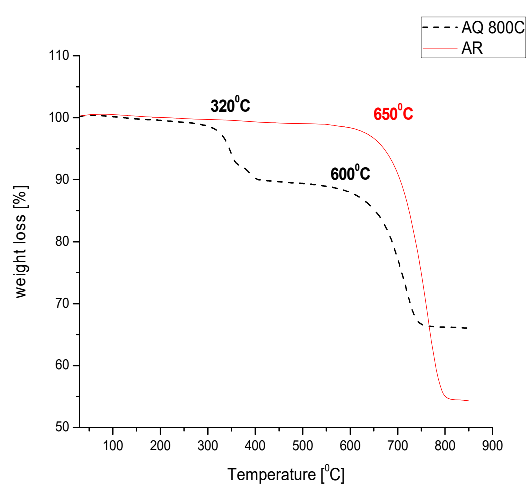


The typical transmittance FTIR spectra of dolomite shows asymmetric stretching(ν_3) of C-O bond at

1435 cm^{-1}, out of plane bending(ν_2) at 880 cm^{-1}, in-plane bending(ν4) at 728 cm^{-1} modes of the carbonate group are found to be active. Besides the internal modes, ($\nu_2+\nu_4$) combination mode has also been observed at 2524cm^{-1}. H-bonded water is observed at 3421cm^{-1} and also there is a peak at 1011cm^{-1} due to silicates is observed. The characteristics dolomite bands are shifted to 1414, 877 and 714 cm^{-1}, in addition, the weak band due to quartz at 456 cm^{-1} is also visible in the FTIR spectra of 800^{0}C heat-treated dolomite, demonstrating the structural transformation of dolomite to calcite. It is also observed that the transmittance of the peak at 1414cm^{-1} is almost double when compared to the FTIR spectra of the as received sample, due to the release of 50% CO_2 from carbonate group and the sample partially converted into hydroxide i.e. $CaMg(OH)_2CO_3$. Hence a sharp band at 3697cm^{-1} observed due to this hydroxyl group. The characteristic absorption peak for dolomite at 2627cm^{-1} differentiates it from calcite, which is not observed in the calcite. This clearly indicates the structural transformation of dolomite to calcite [3].

3.4 TGA of Natural dolomite and AQ 800°C

Fig.4: TGA of Dolomite.

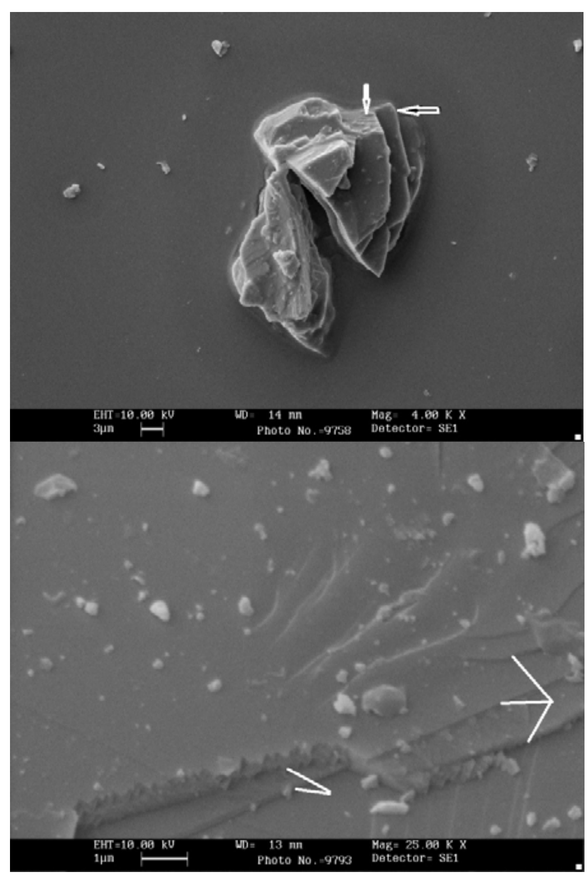


Thermal decomposition of dolomite was studied by TGA from room temperature to 800^{0}C in a nitrogen atmosphere. The typical TGA curve of as received dolomite measured weight loss was 1.33% below 600^{0}C and between 600^{0}C and 800^{0}C reached to 46% which is attributed to the decomposition of carbonates. The TGA of dolomite annealed and quenched from 800^{0}C shows two decomposition bands, one is from 300^{0}C to 400^{0}C and the other is from 600^{0}C to 750^{0}C. It is observed from the first decomposition band the sample lost weight by 11% which may be due to retained water during the recrystallization of a mineral when it is stored after annealed and quenched from 800^{0}C. In the second decomposition band, sample lost weight by 23% which may be due to regain of CO_2 either from $CaCO_3$ or $MgCO_3$ and converted into $CaMg(OH)_2CO_3$ [4].

The SEM micrographs of natural dolomite show the fine layered structure and it is observed that the sizes of these layers are in nanoscale (<1µm). The white spots found on the surface may be due to clusters of SiO_2.

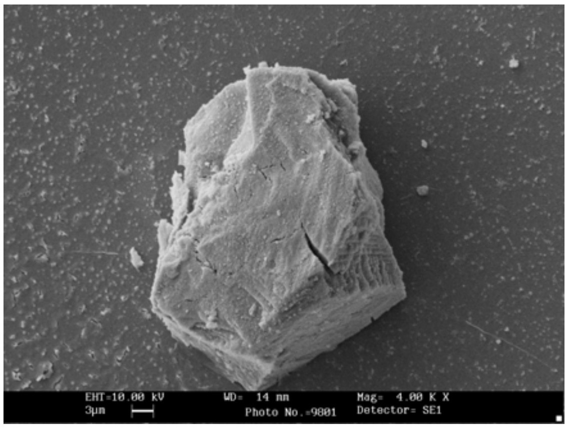
3.5 SEM micrographs of Natural dolomite and AQ 800°C

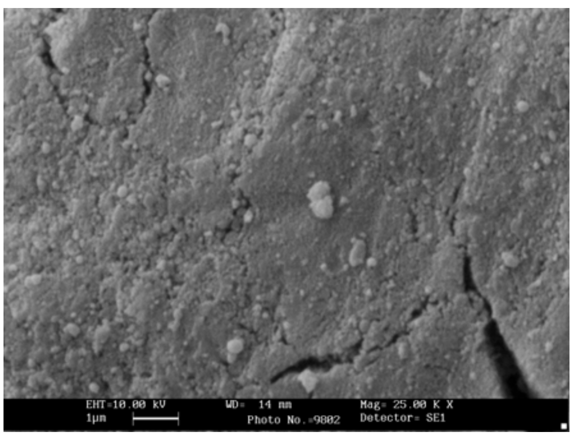
Fig.5:- SEM micrographs of natural dolomite.



When mineral annealed and quenched from 800^{0}C, here surface developed cracks and also an agglomeration of particles due to some deformation is observed.

Fig.6: SEM micrographs of dolomite AQ 800^{0}C





4. Conclusion

The samples annealed and quenched from 200 ^OC, 400 ^OC and 800 ^OC are compared for TL, It is found the intensity increases and TL peak temperature variations are observed. When the sample annealed quenched from 600 ^OC causes a collapse 60% of the sensitivity to ionizing radiation which attributes to the TL property of calcite [5], this also may be due to various phases of MgO, CaO, $CaMg(CO_3)_2$, $CaMgCO_3(OH)_2$ and also the electric dipole and magnetic dipole did not allow to form traps during irradiation. Unstable traps must have been formed due to crystal field effects of various constituents of minerals mixture at 600 ^OC, leads the TL intensity reduced drastically.

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