Piezonuclear Reactions during Mechanical Tests of Basalt and Magnetite

A. Manuello[1a], R. Sandrone[2b], S. Guastella[3c],
O. Borla[1d], G. Lacidogna[1e], A. Carpinteri[1f]

[1]Politecnico di Torino, Dept. of Structural Engineering & Geotechnics,
[2]Politecnico di Torino, Dept. of Environment, Land and Infrastructure Engineering,
[3]Politecnico di Torino, Dept. of Applied Science and Technology,
[a]amedeo.manuellobertetto@polito.it, [b]riccardo.sandrone@polito.it, [c]salvatore.guastella@polito.it,
[d]oscar.borla @polito.it, [e]giuseppe.lacidogna@polito.it, [f]alberto.carpinteri@polito.it

Keywords: Neutron Emission, X-Ray Spectroscopy, Geochemical Evolution.

Abstract. Neutron emissions (NE) were measured during laboratory compression tests of magnetite specimens, loaded up to failure under monotonic displacement control, and of basaltic rocks during cyclic loading tests. In order to detect neutron emissions, the experiments were monitored by two different neutron detectors: He^3 proportional counter and thermodynamic (bubble) detectors. After the tests, Energy Dispersive X-Ray Spectroscopy (EDS) analyses were conducted to recognize possible direct evidences of low energy nuclear reactions (piezonuclear reactions) on fracture surfaces. In particular, quantitative evidences of nuclear reactions, involving Fe decrease and the increases of lighter elements, have been observed in olivine, a crystalline phase widely diffused in the basalt chemical composition. These results confirm similar evidences previously observed on Luserna stone (granite) and confirm that piezonuclear reactions take place in iron-rich materials.

Introduction

It is possible to demonstrate experimentally that the brittle fracture in solid materials is accompanied by the release of different forms of energy [1-3]. In recent studies it has been observed that quasi-brittle materials such as granitic gneiss (Luserna stone) subjected to compression tests under monotonic displacement control, under cyclic loading, and by ultrasonic vibration, are characterized by neutron emissions up to one order of magnitude greater than the background level [1-5]. These tests have been conducted on Luserna stone specimens with different shapes and dimensions and characterized by a Fe content of about 1.5%. It has been observed that, in the case of this rock, the iron oxides are prevalently concentrated into two crystalline phases: phengite and biotite. These two minerals, quite common in the stone (20% and 2%, respectively), have shown important changes in the mineral chemistry of fracture surfaces [6]. In particular, considering the abundances of phengite and biotite in the Luserna stone composition, the reduction in the Fe content (~25%) seemed to be almost perfectly counterbalanced by an increase in Al, Si, and Mg concentrations [6].

In the present paper, neutron emission measurements, by means of He^3 proportional counter and thermodynamic bubble detectors, were performed during compression tests on magnetite specimens and under cyclic loading tests on basalt rocks. The employed materials have been chosen in order to correlate the increasing iron contents (~15% for basalt and ~72.5% for magnetite) with the neutron emissions levels measured during fracture and fatigue tests.

Fracture tests of magnetite were performed using cylindrical specimens of different diameters coming from the San Leone mine. This deposit is located about 30 km southwest of Cagliari, Sardinia (Italy), near the Basso-Sulcis batholit, a granodioritic intrusive of Hercynian age [7]. This mine is almost recent - the occurrence was discovered in 1860 during the development of industrial age which needed always more prime matters and increasing quantities of minerals [7]. In 1892 the mine started its activity, which ended in 1963. The mineralization of the deposit is mainly represented by magnetite. The ore bodies within skarn generated by the thermometamorphism of previous paleozoic limestones at contact with granitic intrusions (300×10^6 years ago). The mean iron concentrations of the San Leone magnetite is between 72.5% and 75% [7]. Another iron-rich material used for the cyclic loading experiments is basalt comes from the mount Etna bacine. Basalt is a very common extrusive igneous rock, is the dominant material making up the Earth's oceanic crust and represent the principal production of volcanoes during their eruptions. The basalt, also called Lava stone, used into the piezonuclear tests, is characterized by a mafic chemistry (with a higher content of iron and magnesium), is dark grey and shows porphyritic texture with few mm-sized phenochrists of plagioclase, clinopyroxene and olivine [8].

After the experiments basalt fracture surfaces were analyzed by Energy Dispersive X-ray Spectroscopy (EDS) in order to evaluate direct evidence of anomalous nuclear reactions that could have taken place during the tests. These results were used to confirm the evidence observed in the case of Luserna stone and to recognize different piezonuclear reactions induced in natural non radioactive rocks characterized by very high iron concentrations. In addition, these results of chemical changes in basalt and magnetite could be used to give a valid interpretation of the different chemical composition between the oceanic and the continental crust. Finally, this evidence may be used also to evaluate the transition from basaltic to sialic crust chemical composition that characterized the geochemical evolutions of the Earth's Crust [9].

Experimental Set-up

Experimental compression tests were performed on brittle magnetite specimens under monotonic displacement control and basaltic specimen under cyclic loadings. All the experimental tests were performed at the Fracture Mechanics Laboratory of the Politecnico di Torino. In particular, in this work the experimental results of four specimens P1, P2, P3 (magnetite) and P4 (basalt) are reported. The tested materials, the shapes and sizes of the specimens, the characteristics of the test types are summarized in Table 1.

Table 1. Tested materials and Test types

Monotonic Displacement Control							
Specimen	Material	D (mm)	H (mm)	λ=H/D	Velocity (mm/s)	Load (kN)	
P1	Magnetite	40	40	1	0,001	197,97	
P2	Magnetite	90	90	1	0,002	932,38	
P3	Magnetite	20	20	1	0,0005	45,05	
Cyclic Loading							
Specimen	Material	D (mm)	H (mm)	λ=H/D	Frequency (Hz)	Maximum Load (kN)	Minimum Load (kN)
P4	Basalt	80	160	2	2	350	30

The fracture experiments on magnetite specimens were performed by uniaxial compression using a MTS servo-controlled hydraulic testing machine with a maximum capacity of 1000 kN. Each test was performed in piston travel displacement control by setting a constant piston velocity between 5×10^{-4} and 2×10^{-3} mm/s. The test specimens were arranged in contact with the press platens without any coupling materials, according to the testing modalities known as "test by means of rigid platens with friction". The fatigue test on the basalt specimen was performed up to the specimen failure at a frequency of 2 Hz with a maximum load of 350 kN and a minimum load equal to 30 kN (Tab. 1).

Experimental Results: Neutron Measurements

The NE measurements on magnetite specimens were performed using an He^3 neutron detector switched on at least one hour before the beginning of each compression test, in order to reach the thermal equilibrium of electronics, and to make sure that the behaviour of the device was stable with respect to intrinsic thermal effects. The average measured background level was ranging from $(4.00\pm1.00)\cdot10^{-2}$ to $(6.40\pm1.60)\cdot10^{-2}$ cps. Neutron measurements of specimen P3 yielded values comparable with the ordinary natural background, whereas in specimens P1 the experimental data exceeded the background value by about five times. For specimen P2 the neutron emissions achieved values of about three orders of magnitude higher than the ordinary background (Fig. 1a). Furthermore, during the compression tests a rise of the thermal equivalent neutron dose, analysed by neutron bubble detectors [10], consistently with the increment of the neutron level measured by the He^3 device, was observed. In particular for the specimen P2, a value of more than 1000 times higher in comparison with the ordinary background was found at the end of the test.

Specimen P4, made of basaltic rock, was subjected to fatigue cycles up to the final failure as summarized in Tab. 1. Droplets counting was performed every 12 hours and the equivalent neutron dose was calculated [10]. In the same way, the natural background was estimated by means of the two bubble dosimeters. During this test the ordinary background was found to be (53.76±13.44) nSv/h. An increment of about twenty times with respect to the background level was detected at specimen failure (Fig. 1b). No significant variations in neutron emissions were observed before the failure. The equivalent neutron dose, at the end of the test, was (935.49±233.87) nSv/h.

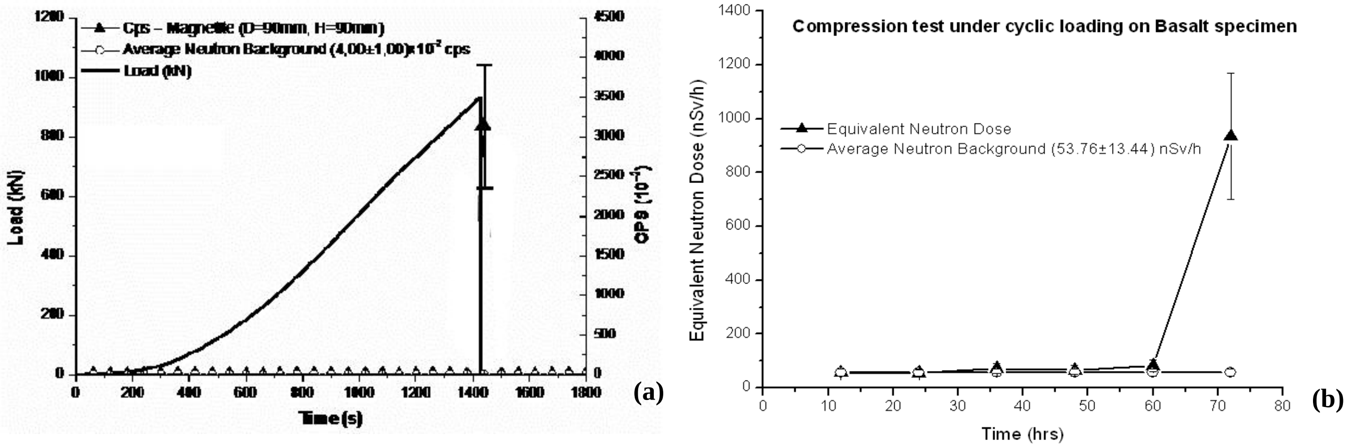

Fig. 1. (a) Magnetite: For specimen P2 the neutron emissions achieved values of about three orders of magnitude higher than the background level. (b) Basalt: An increment of about twenty times in comparison with the background level was detected at the failure of specimen P4 (fatigue test).

Eds Analysis on Basalt

After the mechanical loading experiments, Energy Dispersive X-ray Spectroscopy (EDS) was performed on different samples of external and fracture surfaces, belonging to the same specimens used during the cyclic loading test (basalt). The analysis were conducted in order to correlate the neutron emission from the specimen with the variations in rock composition due to the mechanical loading. These analyses lead to get averaged information of the mineral chemical composition and to detect possible anomalous transformation from iron to lighter elements. The quantitative elemental analyses were performed by a ZEISS Supra 40 Field Emission Scanning Electron Microscope (FESEM) equipped with an Oxford X-rays microanalysis [7].

The first analysis were performed on fracture surfaces of basalt specimen P4 after the fatigue test and the consequent failure. In this case, similarly to the case of Luserna stone [7], taking into account the heterogeneity of the material, the samples were carefully chosen to investigate and compare the same crystalline phase before and after the failure. In particular, olivine, was considered due to its high iron content (olivine Fe content ~24%) and because it is quite common into this type of rock [7]. In the case of basalt two different kinds of samples were examined: (i) polished thin sections, finished with a standard petrographic sample procedure, covered by Cr, for what concerns the external surface of the basalt; (ii) small portions of fracture surfaces without any kind of preparation, apart from the Cr covering, for what concerns the fracture surface of the same material. Semi-quantitative standardless analysis was performed on the collected spectra, fixing the stochiometry of the oxides, to correlate the oxides content with the specific crystalline phase. The Cr lines were excluded from the semi-quantitative evaluation [7].

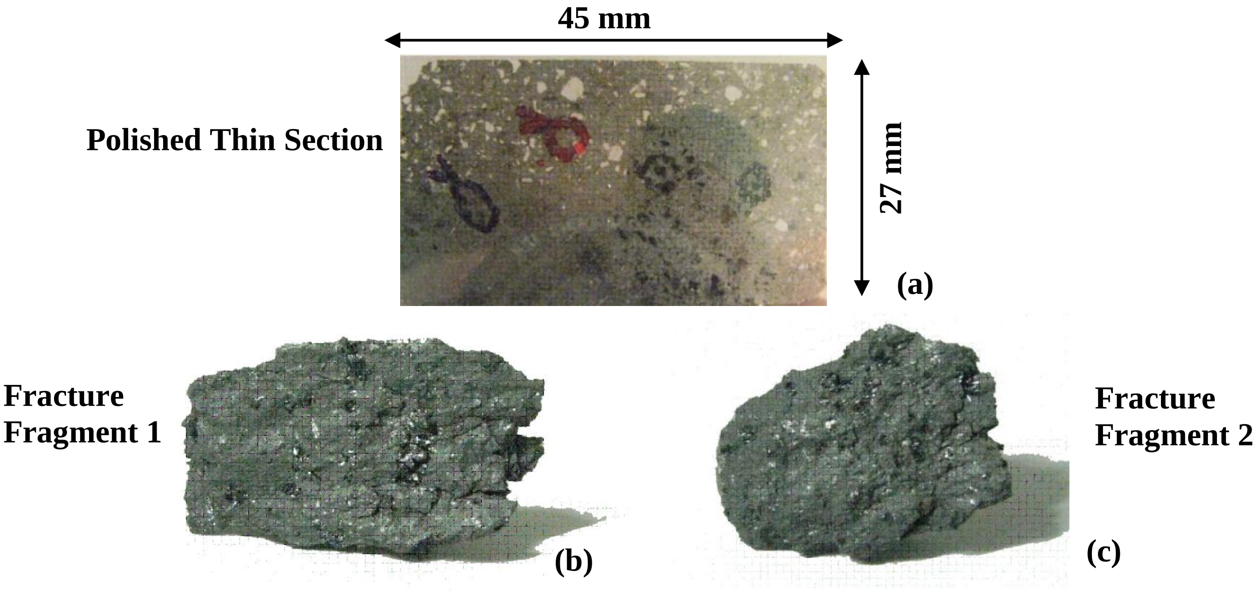

Fig. 2. (a) Polished thin sections, finished with a standard petrographic sample procedure, covered by Cr, were examined to evaluate olivine composition on external surface; (b,c) small portions of fracture surfaces without any kind of preparation, apart from the Cr covering, were analysed to evaluate the chemical changes in olivine on fracture surface.

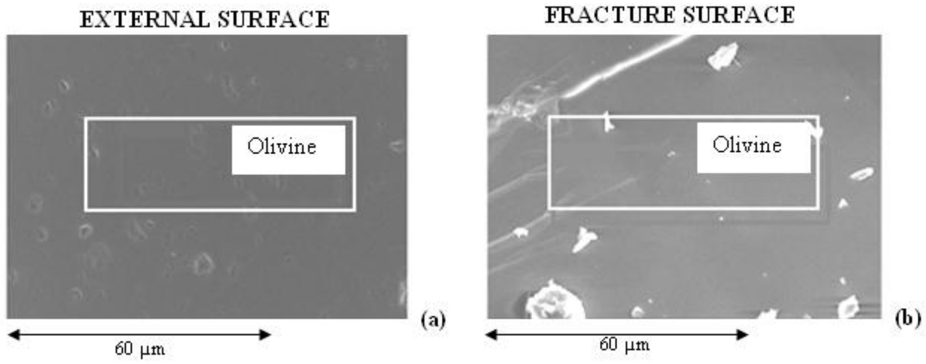

Fig. 3. Field emission scanning electron microscope images of olivine in the case of (a) external and (b) fracture sample.

In Figure 2a, a polished thin section obtained from the external surface of an integer and uncracked portion of the basalt specimen P4 is shown. The polished thin section presents a rectangular geometry (45 × 27 mm) and is 30 μm thick. This kind of analysis involves thousands of cubic microns for each acquisition area in the investigated sample. From this point of view there exists a substantial difference between these analysis and the spot analysis reported for the Luserna stone samples [7]. In this case, in fact, a larger portion of material in involved and each analysis is indicative of an investigated volume equal to $60\times20\times4\ \mu m^3$ (see Fig 3a and 3b).

In Fig. 4a,b,c the distributions of Fe, Si and Mg concentrations are reported for external and fracture surface of specimen P4. It can be observed that the distribution of Fe content for the external surfaces shows an average value equal to 18.40% (Fig 4a). In the same graph the distribution of Fe concentrations on the fracture samples shows significant variations. It can be seen that the mean value of the distribution of measurements performed on fracture surfaces is equal to 14.40% and it is considerably lower than the mean value of external surface measurements (18.40%). Similarly to Fig. 4a, in Fig 4b the Si mass percentage concentrations are considered. For Si contents, the observed variations show a mass percentage increase approximately equal to 2.20%. The average value of Si concentrations changes from 18.30% on the external surface to 20.50% on the fracture surface. In Fig. 4c it is shown that, in the case of olivine, also Mg content presents considerable variations. Fig. 4c shows that the mass percentage concentration of Mg changes from a mean value of 21.20% (external surface) to a mean value of 22.80% (fracture surface) with an increase of 1.60%. Therefore, the iron decrease (–4.00%) in olivine seems to be almost perfectly counterbalanced by an increase in silicon (+2.20%), and magnesium (+1.60%). In addition, other analysis conducted on olivine localized on the fracture surface have shown the appearance of Al_2O_3 into the chemical composition of this crystalline phase (see Fig. 5). This evidence seems to be particularly important because no Aluminium traces were observed in olivine sample localized on the external surface of the same rock. This fact represents a further confirmation that in basaltic olivine similarly to the case of phengite in Luserna stone [7], the following peizonuclear reaction occurred:

$$\mathrm{Fe}_{26}^{56} \rightarrow 2\mathrm{Al}_{13}^{27} + 2\ \text{neutrons} \qquad (1)$$

At the same time, the results involving the Fe decrease and the consequent increases of Si and Mg, discussed above and reported in Fig. 4, lead to the conclusion that also in olvine, in addition to biotite in Luserna stone [7], the following piezonuclear reaction occurred during the mechanical loading:

$$\mathrm{Fe}_{26}^{56} \rightarrow \mathrm{Mg}_{12}^{24} + \mathrm{Si}_{14}^{28} + 4\ \text{neutrons}. \qquad (2)$$

The results reported in Figs. 4 and 5 represent also an important significance from a geophysical point of view. In fact, at the scale of the Earth’s crust, the non-homogeneous composition of oceanic crust and continental crust could be explained by the transition from basaltic to sialic crust composition. At the sea bottom, the eighty-five percent of the Earth’s volcanic eruptions take place in correspondence to mid ocean-ridges [11]. These submarine volcanoes generate the solid underpinnings of all the Earth’s oceans (oceanic crust) [12-16]. Comparing the data presented in the literature concerning the composition of the two different types of terrestrial crust, it can be noted that the iron concentration changes from ~8 %, in the oceanic crust, to ~4 % in the continental one. Ni changes from ~0.03 %, in the oceanic crust to ~0.01 % in the continental one (about a three-fold decrease). And vice versa, Al, Si, Mg and Na vary from ~7%, ~24%, ~3.6% and ~1% in the oceanic crust, to ~8 %, ~28 %, ~1.3% and ~2.9% in the continental crust, respectively. Considering that approximately 50% of the continental crust has originated over the last 3.8 Gyrs, as the result of oceanic crust subduction [3-5,9,11-16], the results presented in this paper could be used to offer a further confirmation that piezonuclear reactions are a valid explanation for the chemical changes of the crust in correspondence of mechanical phenomena of fracture, crushing, fragmentation, comminution, erosion, friction, during seismic events [3-5,9].

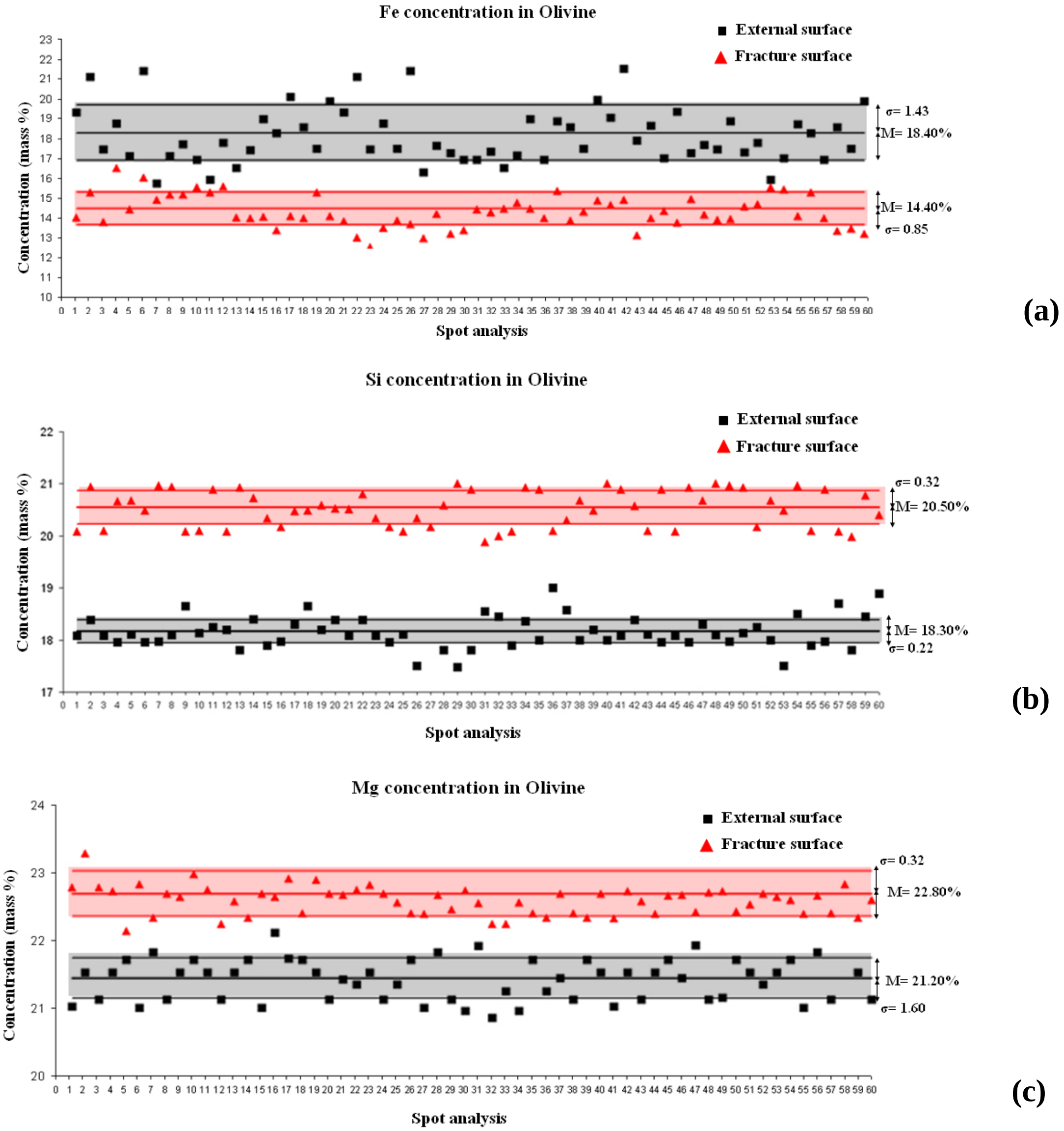

Fig. 4. Olivine chemical changes after mechanical loading: (a) The Fe decrease (–4.00%) is almost perfectly counterbalanced by an increase in Si (b) (+2.20%), and Mg (c) (+1.60%).

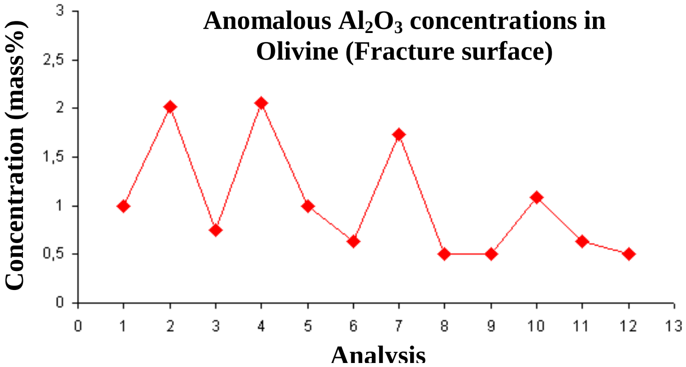

Fig. 5. Al_2O_3 appeared in the olivine composition after the mechanical loading experiments (fracture surface).

Conclusions

We report the results of energy emissions in the form of neutron bursts measured during the application of monotonic displacement and cyclic mechanical loading on Sardinian magnetite and Etna basalt specimens respectively. These analyses are strictly connected with recent results obtained in similar tests using inert rocks with a lower Fe content and more heterogeneous mineral compositions (Luserna Stone).

The investigation confirms that the pressure, suitably exerted on inert medium of stable nuclides, generates nuclear reactions of a new type (piezonuclear reactions) with a substantial chemical changes into the mineral compositions. EDS analysis was performed on different samples of external or fracture surfaces belonging to basalt specimens in order to get averaged information about possible changes in the chemical composition. Considering the results for olivine a considerable reduction in the iron content (–4%) is counterbalanced by the increases in lighter elements such as Si, and Mg. These results together with the appearance of Al in the form of Al_2O_3 into the chemical composition of olivine localized on the fracture surface are significant evidence that piezonuclear reactions (1) and (2) take place in basalt rocks in addition to granite. Finally, the hypothesis of piezonuclear reactions seems to find surprising evidence and confirmation at the Earth crust scale. The piezonuclear reactions have thus been considered in order to interpret the most significant geophysical and geological transformations, today still unexplained.

Acknowledgements

The financial support provided by the Regione Piemonte (Italy) RE-FRESCOS Project, is gratefully acknowledged. Special thanks are due to Mr. F. Argiolas for the providing of the magnetite specimens.

References

[1] A. Carpinteri, F. Cardone and G. Lacidogna: Strain Vol. 45 (2009), p. 332.
[2] F. Cardone, A. Carpinteri and G. Lacidogna: Physics Letters A Vol. 373 (2009), p. 4158.
[3] A. Carpinteri, F. Cardone and G. Lacidogna: Experimental Mechanics Vol. 50 (2010), p. 1235.
[4] A. Carpinteri, O. Borla, G. Lacidogna and A. Manuello: Physical Mesomech. Vol. 13 (2010), p. 268.

[5] A. Carpinteri, G. Lacidogna, A. Manuello, O. Borla: Strenght Fracture and Compl. Vol. 7 (2011), p. 13.

[6] A. Carpinteri, A. Chiodoni, A. Manuello and R. Sandrone: Strain. Vol. 47**,** (2011), p. 282.

[7] J. Verkaeren, P. Bartholomè: Economic Geology Vol 74 (1979), p. 53

[8] J. C. Tanguy: Contributions to Mineralogy and Petrology Vol. 66 (1978), p. 51.

[9] A. Carpinteri and A. Manuello: Strain. Vol. 47**,** Suppl. 2, (2011), p. 267.

[10] Bubble Technology Industries. Chalk River, Ontario, Canada, 1992.

[11] C.D. Catling and K.J. Zahnle: Scientific American Vol. 300 (2009), 24

[12] S. R. Taylor and S. M. McLennan: Reviews of Geophysics Vol. 33 (1995) p. 241.

[13] S.R. Taylor, and S.M. McLennan: *Planetary Crusts: Their Composition, Origin and Evolution*, Cambridge University Press, Cambridge, (2009).

[14] C.M.R. Fowler: *The Solid Earth: An Introduction to Global Geophysics*, Cambridge University Press, Cambridge, (2005)

[15] C. Doglioni in: *Treccani, Enciclopedia Scienza e Tecnica* (2007), p. 595.

[16] R.L. Rudnick, D.M. Fountain: Rev. of Geophys. Vol. 33 (1995), p. 267.