Convénio Portugal (FCT) / Itália (CNR) Procº 441.0
PROCESSOS LUMINESCENTES-DOSIMETRICOS NO QUARTZO
Relatório Técnico/Financeiro Anual, Anexo C

Results of Initial Tests

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Initial tests have been carried out on quartz refined from two granite and two pegmatite samples.

The parent materials exhibit similar levels of most elements analysed by neutron activation analysis (Fig 1). However, the granites exhibit much less depletion of Na than the pegmatites, relative to upper continental crust values (Fig 1). The pegmatites are expected to have higher levels of Li than the granites, presently based on the presence of a mineral thought to be spodumene encountered during sample preparation. Refined quartz samples are undergoing analysis, and the Li concentrations remain to be measured. Alkali ions compensating substitutional centres are considered important in the production of luminescence signals in quartz.

The quartz obtained from the granites is transparent and exhibits clean crystal facets little attacked by the acid etching used to purify the samples (Fig. 2). That from pegmatites is milky and exhibits severe acid pitting.

IRSL signals were generally more than 3 orders of magnitude lower than OSL signals (increases following higher anneals may represent phosphorescence, Fig 3). This is indicative of “clean” quartz with few inclusions.

The sensitivity of these geological samples increased little through rapid heating to 500 C, but increased dramatically for 1 hr anneals up to 800 C. For anneals of 1000 and 1100 C some decreases were observed, but other signals continued to increase monotonically (Fig 4). This contrasts with previous findings for archaeological ceramics, where rapid heating to 500 C produced a strong increase that was erased by moderate annealing, before being recovered by higher temperature anneals. This difference is indicative of long- and short- term dosimetric effects following crystallization or heating, and may be important in the understanding of sample behaviours for dosimetry and dating. It indicates that heated samples should be included in the study as it progresses.

One of the pegmatite samples exhibited very low OSL and TSL signals, but the other pegmatite exhibited relatively stronger TSL emissions than the granites at high temperatures and in the blue-green, compared to the low temperature and UV-blue respectively (Fig 5). The TSL and OSL emissions from the pegmatite were less strongly sensitized by moderate temperature anneals than the granites (Fig 4, Fig 6). For higher temperature anneals a TSL peak around 150 C sensitized more strongly than the habitually observed “110 C peak” (Fig 4, Fig 6): in the pegmatite it came to dominate, but in the granites remained subsidiary. The differences in emission wavelength, the behaviour of the 150 C TSL peak, and the temperature of the onset of sensitization, appear to be useful areas to explore in relation to differences in alkali ion content (Na vs. Li) in these quartzes.

Radioluminescence indicated broad blue-green emissions from the granites and narrower blue emissions from the pegmatites, both of which decreased in intensity with increasing dose. This contrasts with the relative intensities of the TSL bands measured from these samples: the radioluminescence spectra were measured at room temperature so the difference may indicate temperature dependent quenching or shifts in emission wavelength. Elevated temperature RL measurements may therefore be important to understand TSL behaviour. A weaker emission centred around 620 nm (red) was observed in all samples. This did not respond to dose, but does appear to relate to grain-surface defects. A single large pegmatite grain was measured whole and cleaved: the freshly cleaved interior yielded a much lower emission intensity for this peak.

Ionoluminescence indicated a more complex series of emissions (Fig 8). A peak around 650-700 dominates the spectra and may relate to the red RL emission. This is relatively higher for the pegmatite than the granite, which would be consistent with the surface effects discussed above. The main RL/TSL emission band in the range 400-500 nm appears relatively smaller, but divided into three separate peaks. RL indicates that this emission region is radiation sensitive, so the relatively low IL intensity may relate to severe quenching under proton irradiation (a potentially useful effect). Two entirely different emission peaks are also evident around 340 nm and 760 nm. IL appears to offer a high-resolution means to investigate luminescence emissions from these samples, but the experimental set-up and data processing still require optimization

Instrumental Neutron Activation Analysis (INAA)

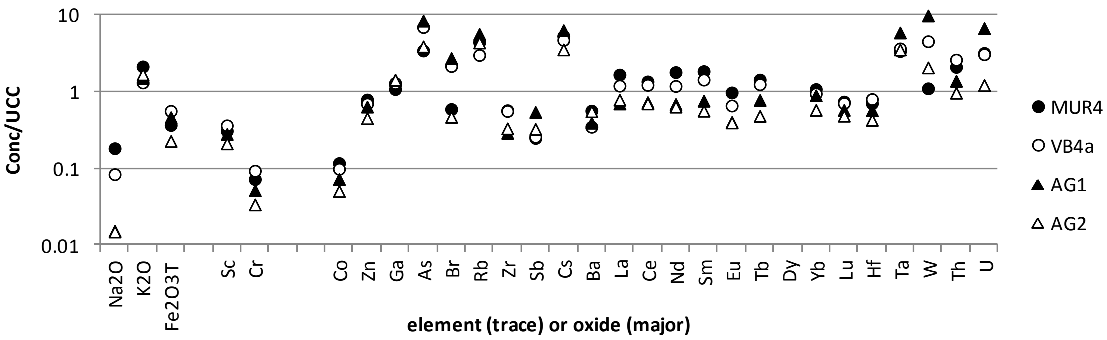

Fig. 1. Elemental concentrations in the parent material from which the quartz was refined, normalised to upper continental crust values.

Thermoluminescence (TSL), Optically Stimulated Luminescence (OSL), and Infrared Stimulated Luminescence (IRSL)

Hand picked quartz grains
AG1 - Pegmatite
AG2 - Pegmatite
VB4a - Granite
MUR4 - Granite

1. Test TSL (240°C@5°C/s), IRSL, OSL and TSL (500°C@5°C/s) natural signal and response to 0.7 Gy and 35 Gy beta radiation doses (also to sensitise sample)

2. 35 Gy beta, TSL repeated with different filter combinations:
UV/Blue (to 500°C)
Blue/Green (to 450°C)
Yellow/Orange (to 400°C)
Red (to 350°C)
(U340 repeat to check for sensitisation)

Repeat 1. and 2. for pre-anneals:
a. 500°C TSL only
b. 600°C/1hr
c. 800°C/1hr
d. 1000°C/1hr
e. 1100°C/1hr

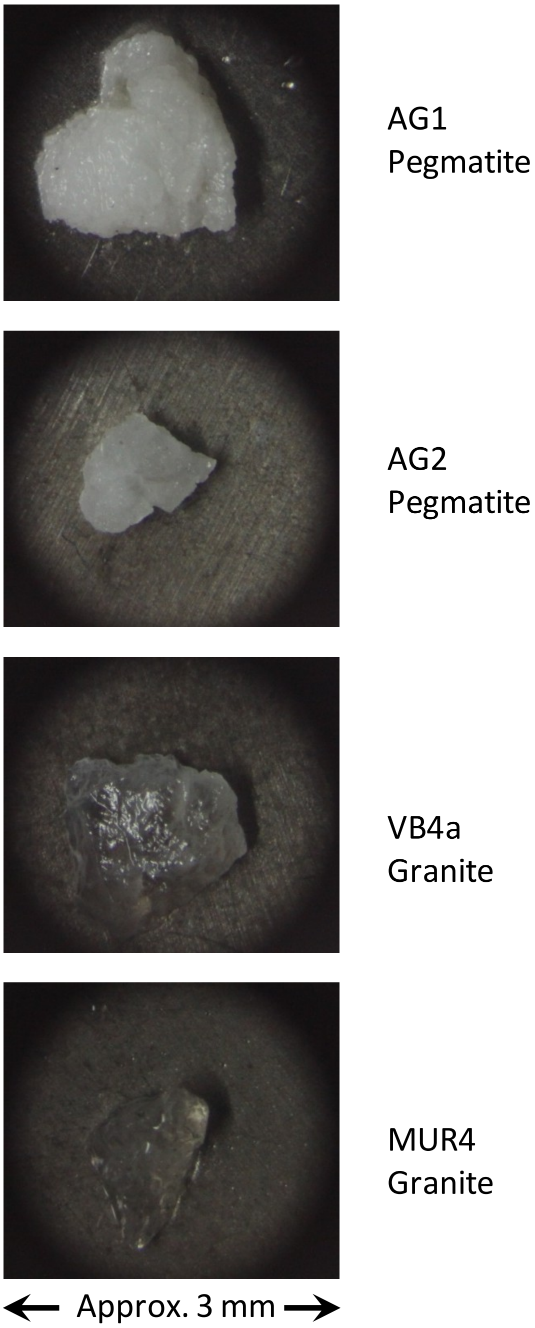

Fig. 2. Samples measured in initial luminescence tests

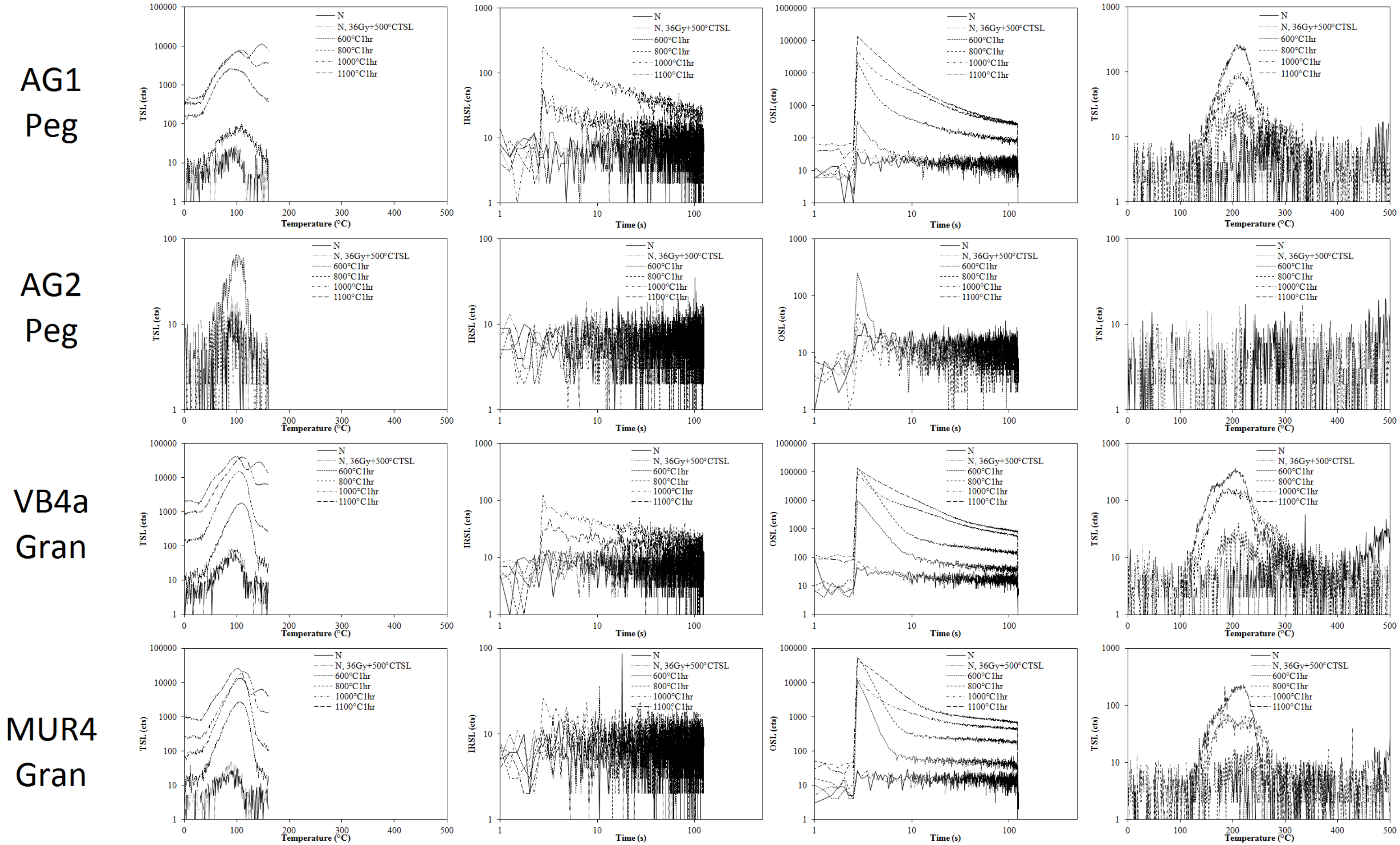

Fig. 3. 0.7 Gy responses of TSL (to 160 °C), and subsequently measured IRSL, OSL and high temperature TSL, from Steps 1. a,b,c,d,e.

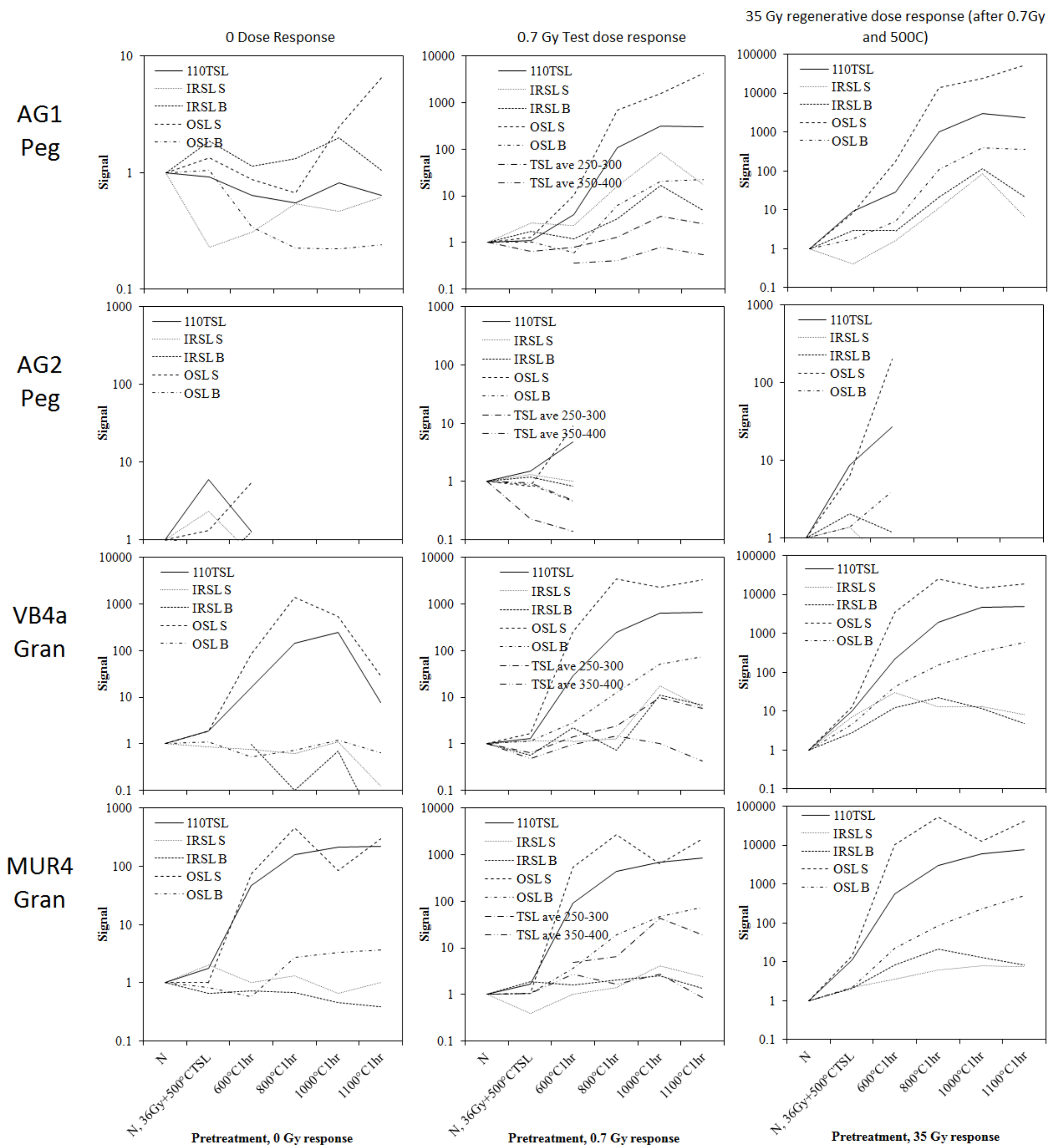

Fig. 4. Changes in integral signals as a function of pretreatment. From Steps 1. a,b,c,d,e

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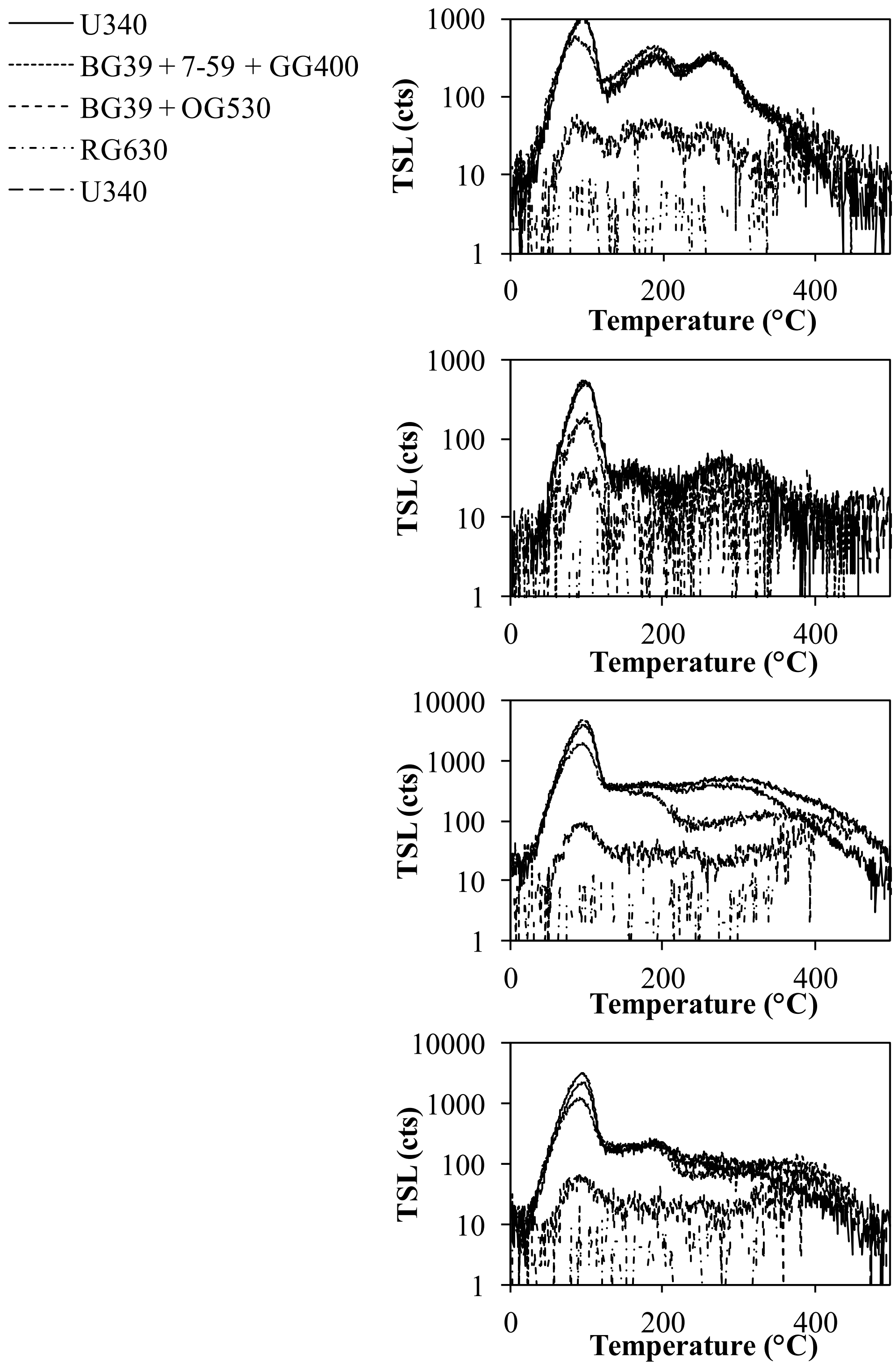

Fig 5. 35 Gy TSL responses in different detection bands. Step 2 a

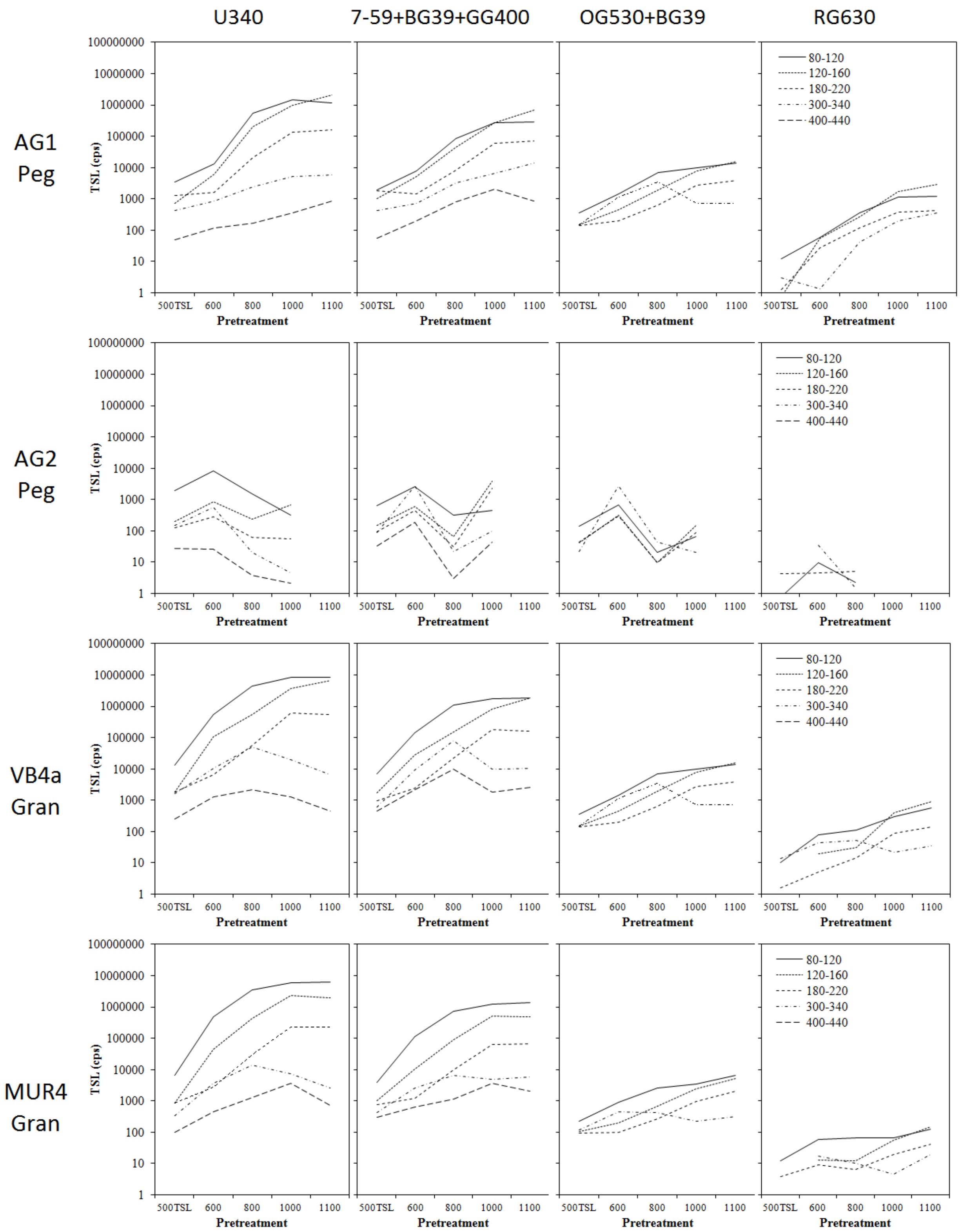

Fig. 6. Changes in integral signals as a function of pretreatment. Steps 2 a, b, c, d, e.

Radioluminescence (RL) and Ionoluminescence (IL)

Radioluminescence spectra were measured in the CUDaM laboratories at UNIMIB, Italy. Emission spectra were measured under irradiation with a 20 kV X-ray source. Repeat measurements were made of each subsample to examine differences in the decrease in RL of different emission bands, as a function of increasing dose (quenching).

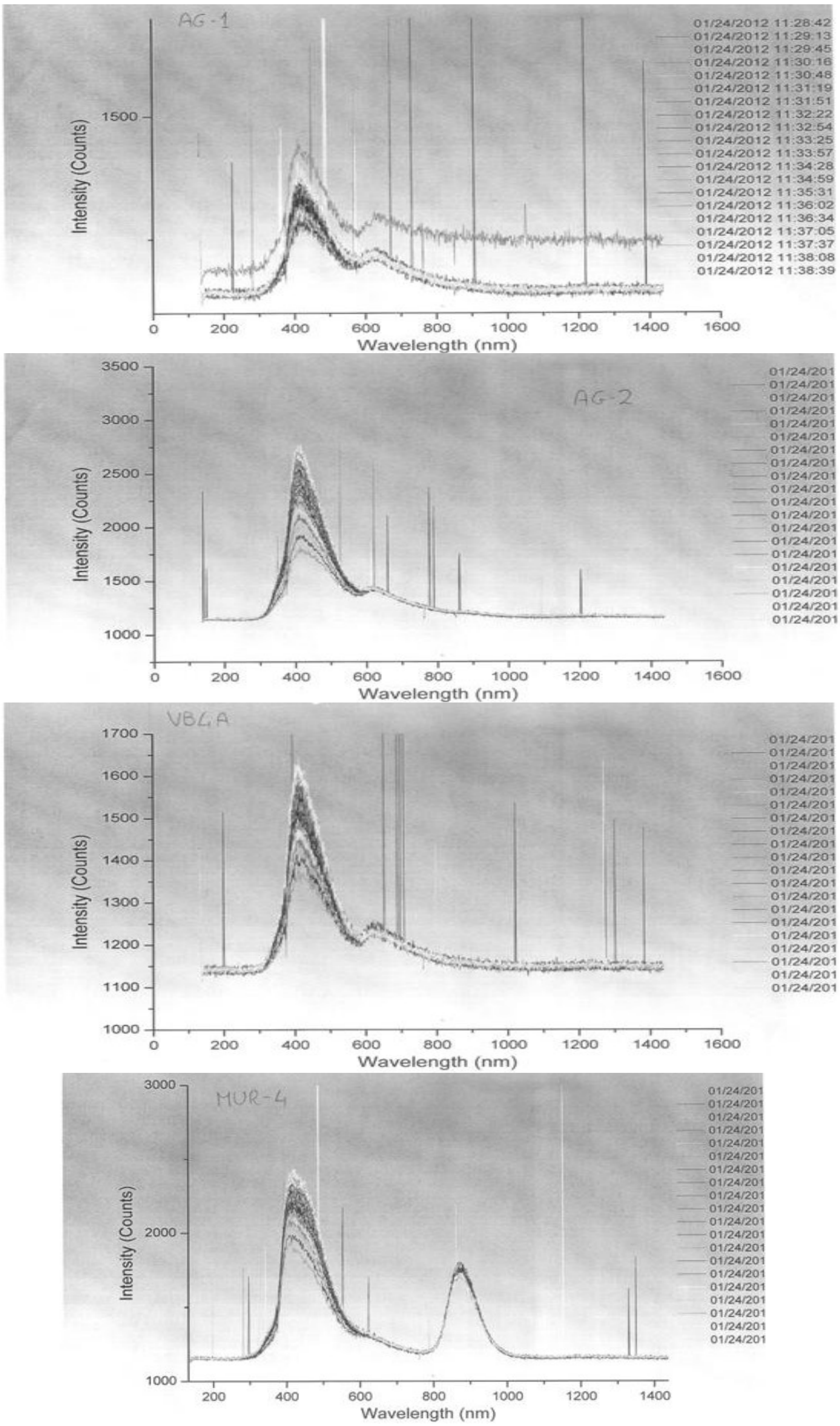

Fig. 8. Radioluminescence spectra from repeated 20 kV photon irradiation. The large peak around 840 nm in the plot for MUR4 is thought to be an artefact.

Ionoluminescence spectra were measured in the microbeam facility of the UFA, IST/ITN by L. Alves, and with the colaboration of V. Corrigidor. Emission spectra were measured under irradiation with a 1 MeV proton beam. Spectra were collected after the samples had been exposed to significant doses of protons, so that the radiation sensitive emission bands had been quenched.

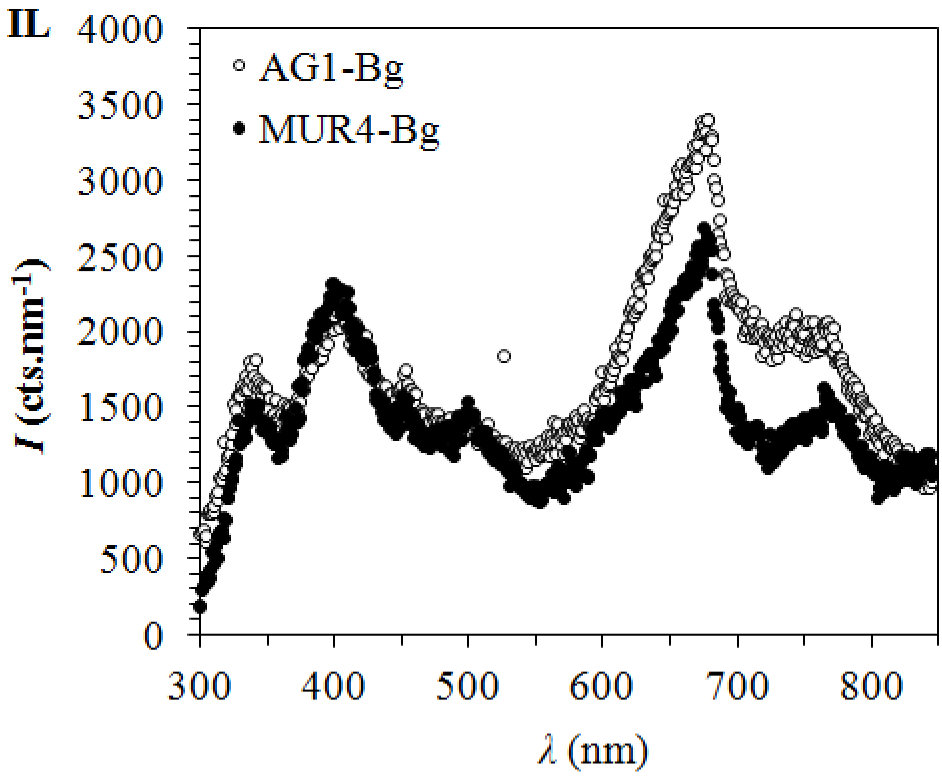

Fig. 8. Ionoluminescence spectra from 1 MeV proton irradiation of one pegmatite and one granite sample.