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Assessing the effectiveness of the double-SAR protocol in isolating a luminescence signal dominated by quartz

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Abstract

The effect of the presence of feldspars that can be shown to exhibit anomalous fading is investigated using the double-SAR protocol. This protocol has been suggested as a method of isolating an optically stimulated luminescence (OSL) signal dominated by quartz from a polymineralic mixture of grains. The progressive removal of feldspars from the silt-sized fraction of sediments from Bignell Hill, Nebraska, is also investigated using chemical treatment with hydrofluorosilicic acid for different durations; the appropriate treatment duration for these sediments is identified as being 7 days. For untreated material, the double-SAR protocol yields IRSL and [post-IR] OSL signals that are both dominated by feldspars and that give the same value for the equivalent dose (D_e) determination. However, for material treated with hydrofluorosilicic acid for 7 days, the IRSL and [post-IR] OSL signals have distinctly different characteristics and appear to be dominated by feldspar and by quartz, respectively. Furthermore, the D_e values determined for the feldspar-dominated IRSL signal are about half those obtained for the quartz-dominated [post-IR] OSL signal. This demonstrates that the double-SAR protocol is not always capable of isolating a quartz-dominated signal; clearly, in some circumstances it is necessary to apply a chemical pre-treatment to remove the contribution from feldspars prior to any luminescence measurements being made.

Keywords: Double-SAR; Quartz and feldspar; Anomalous fading; Hydrofluorosilicic acid etching; Loess; Fine and coarse silt grains

1. Introduction

Access to loess deposits containing feldspars of volcanic origin has provided an opportunity to exploit the different optically stimulated luminescence (OSL) characteristics of quartz and feldspar in order to examine methods of separating the luminescence signals from these two minerals, and to determine the effect of progressive feldspar removal on OSL signals and the resultant equivalent dose (D_e) determinations. The loess deposit investigated is located in the central Great Plains of North America, at Bignell Hill, Nebraska. Geochemical studies of the Peoria (last-glacial) loess at Bignell Hill show that it is derived principally from the Tertiary White River Group sediments that outcrop in eastern Colorado and Wyoming, western Nebraska, and southwestern South Dakota (Aleinikoff et al., 1998, 1999; Muhs et al., 1999, in press), and contains significant quantities of volcaniclastic feldspars. The presence of such material in these deposits is clearly a concern given the problems frequently noted for anomalous fading of the feldspar signal, particularly for volcaniclastic feldspars (Wintle, 1973; Spooner, 1994a; Huntley and Lamothe, 2001). One means of circumventing this problem is to undertake OSL dating of these loess deposits using the signal from quartz.

Some previous studies (e.g., Banerjee et al., 2001; Roberts et al., 2001; Thomas et al., 2003; Stokes et al., 2003; Watanuki et al., 2003) have attempted to isolate the luminescence signal from quartz by applying the ‘Double-SAR’ (Single-Aliquot Regenerative dose) measurement protocol to aliquots of mixed-mineralogy (Banerjee et al., 2001; Roberts and Wintle, 2001, 2003). This technique involves stimulation with infra-red (IR) to deplete the luminescence signal from feldspars (Spooner, 1994a; Duller, 1997) prior to stimulation with blue diodes to obtain what is termed the ‘[post-IR] OSL’ signal which should

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enhance the relative contribution from quartz (Short and Huntley, 1992; Spooner, 1994b), having reduced the contribution from feldspars. An alternative approach is to isolate quartz using physical and chemical separation techniques designed to refine mixed-mineralogy material. This paper examines both of these methods as applied to the loess from Bignell Hill, comparing the luminescence signals and equivalent dose (D_e) values observed.

2. Considerations for dating loess deposits

Given the typically fine-grained nature of loess, and also to avoid complications of assessing the alpha-contribution, 'fine grains' (i.e. 4–11 μm silt) are often used for OSL dating of loess deposits. However, the particle-size range and distribution observed can be very variable, meaning that fine-silt sized material is not always representative of the loess deposit as a whole. For some coarse-grained loess deposits, working with a coarser grain size may be preferable, avoiding questions of potential post-depositional movement of fine-grains through the section. In any case, working with a grain size which reflects the modal grain size of the deposit would seem advisable.

In the case of the Bignell Hill sediments, fine-silt (2–20 μm diameter) represents only approximately 10–15% of the loess, and the dominant grain size is coarse-silt (20–53 μm, typically representing ∼ 40% of the loess) (Winspear and Pye, 1995; Mason et al., 2003; Muhs et al., in press). In spite of the relatively coarse nature of the loess it is, however, also possible to recover sufficient material to make measurements on the polymineral fine grain (4–11 μm) fraction. In this paper, both fine-silt and coarse-silt sizes are considered, using material in the ranges 4–11 and 35–50 μm, respectively. The effect of the progressive chemical removal of the feldspathic component is examined.

Fine-grained quartz (defined from this point onwards, as material of less than 50 μm in diameter, unless specified otherwise) can be isolated from sediments using hydrofluorosilicic acid (H_2SiF_6) (Jackson et al., 1976; Berger et al., 1980), or using hydrofluoric acid (HF) (Prasad, 2000; Mauz and Lang, 2004). Hydrofluoric acid is used routinely in the preparation of coarse-grained (i.e., > 90 μm diameter) quartz because it not only dissolve feldspars, but it also removes the outer, alpha-irradiated portion of the quartz grains. For example, Hong (1998) demonstrated that using 40% HF applied for 40 min removes an average of ∼ 10 μm from the outer surface of quartz grains of mean diameter 137.5 μm (assuming spherical grains and isotropic etching). In contrast, when pre-treated with commercially available silica to remove any free hydrofluoric acid, hydrofluorosilicic acid will selectively dissolve aluminosilicates and other non-quartz components, leaving the quartz component intact (Berger et al., 1980). Treatment with hydrofluorosilicic acid therefore offers clear advantages over treatment with hydrofluoric acid for the preparation of fine-grained quartz, because the latter treatment will clearly also etch the quartz grains, reducing the quartz yield. Fine-grained quartz of 4–11 μm size has been successfully isolated through use of hydrofluorosilicic acid in a number of studies (e.g., Wintle, 1982; Rees-Jones, 1995; Zhou and Shackleton, 2001; Wang et al., 2006). In the present study, hydrofluorosilicic acid is used to isolate fine-grained quartz, and an experiment is conducted to determine the optimal etch time to ensure effective removal of feldspar from a polymineralic-mixture.

3. Methods

3.1. Sample pre-treatment

A sample of Peoria loess from Bignell Hill (Aber/44-BH-37) was pre-treated with 20 vol. hydrogen peroxide to remove any organics, and then with 50% v.v. hydrochloric acid to remove carbonates and to remove surficial coatings. The material was then settled over 20 cm depth of 0.01 M sodium oxalate solution for 20 min, to separate the > 11 μm fraction. This fraction was then wet-sieved using nylon mesh of 50, 35 and 20 μm, and the 35–50 μm fraction was subjected to treatment with hydrofluorosilicic acid, according to the method described below.

The < 11 μm fraction was settled twice in 0.01 M sodium oxalate for 4 h over 20 cm depth, to isolate the 4–11 μm fraction according to Stokes' Law settling. Polymineral fine-grain aliquots were prepared by dispensing 1 mg of material suspended in acetone onto 9.7-mm diameter aluminium discs, allowing the acetone to evaporate leaving an even deposit of grains on each disc.

3.2. Hydrofluorosilicic acid treatment

Berger et al. (1980) outlined a procedure for isolation of fine-grained (4–11 μm) quartz using hydrofluorosilicic acid. In that study, the acid was pre-treated with commercially available silica of 4–88 μm size, and stored for at least 3 days at 4 °C prior to use. The commercial silica was centrifuged out of the acid prior to application in the ratio of 40:1 for liquid:solid mass. Treatment was for 6 days for each 50 mg of plagioclase present.

In the present study, refrigerated 35% hydrofluorosilicic acid was pre-treated with a large quartz crystal to remove any free HF, and the acid was centrifuged prior to use to avoid any question of contamination of the acid with added silica. Hydrofluorosilicic acid was added to 35–50 μm diameter sample material in the ratio of 40:1, acid:sediment mass. The volume of acid was monitored and maintained as necessary during the etch period, and samples were agitated twice each day. At the end of the etching period, samples were centrifuged and the hydrofluorosilicic acid decanted. Samples were then treated with concentrated hydrochloric acid solution for 45 min, to break down any fluorides formed during the etching procedure. Finally, samples were centrifuged, the hydrochloric acid was decanted, and samples were then rinsed three times in distilled water prior to being re-sieved at 35 μm.

Five aliquots of 35–50 μm sample material were treated with hydrofluorosilicic acid for varying durations to determine the