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ScienceDirect
Chemical Geology 252 (2008) 21–30
CHEMICAL GEOLOGY
INCLUDING
ISOTOPE GEOSCIENCE
www.elsevier.com/locate/chemgeo

Robotic micromilling of tree-rings: A new tool for obtaining subseasonal environmental isotope records

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Accepted 31 January 2008

Abstract

High-resolution, intra-ring $\delta^{18}O$, δD, and $\delta^{13}C$ values of modern tree species (*Picea glauca*, *Larix laricina*, and *Populus grandidentata*) were obtained using a robotic micromill. The micromilling apparatus permits accurate mapping and sampling of growth bands at a resolution of a few µm, producing a fine, homogenous powder that facilitates chemical processing and analyses of very small aliquots of α-cellulose and cellulose nitrate (~100 µg). Considerable intra-ring isotope variability observed in tree samples in this study correlates with temperature and precipitation data indicating that intra-ring variability in $\delta^{18}O$, $\delta^{13}C$, and δD has great potential for reconstructing high-resolution variation in meteorological conditions throughout the growing season.

Keywords: High-resolution; Intra-ring; Cellulose; $\delta^{18}O$; $\delta^{13}C$; δD

1. Introduction

Isotope studies of tree-ring cellulose (e.g., $\delta^{18}O$, $\delta^{13}C$, and δD analyses) are increasingly used as proxies for terrestrial climate variability (e.g., Epstein et al., 1977; Wilson and Grinsted, 1977; Saurer et al., 1997; Switsur and Waterhouse, 1998; McCarroll and Loader, 2004; Miller et al., 2006; Loader et al., this volume). Isotope time series developed from tree-ring cellulose have advantages over other proxies such as speleothems and lake sediments because they provide precisely dated annual and even subseasonal records. Additionally, developments in online continuous flow gas isotope ratio mass spectrometry technology permit more rapid analysis of increasingly smaller α-cellulose samples (~100 µg). Intra-ring sampling for isotope analyses can provide records of meteorological and atmospheric conditions at monthly or even weekly resolution (Leavitt and Long, 1991; Loader et al., 1995; Evans and Schrag, 2004). High-resolution dendro-isotope studies are particularly useful for generating records of seasonal variability in precipitation source (i.e., monsoonal rain), temperature and relative humidity (Loader et al., 1995; Evans and Schrag, 2004; Poussart et al., 2004; Poussart and Schrag, 2005; Poussart et al., 2006), and recently as a record of tropical cyclones (Miller et al., 2006). Additionally, researchers have used high-resolution sampling to develop records of annual variations in tropical trees where growth banding is not necessarily annual (Evans and Schrag, 2004; Poussart et al., 2004; Poussart et al., 2006).

Traditionally, high-resolution sampling has been conducted by fixed blade and rotary microtomes (Loader et al., 1995; Evans and Schrag, 2004; Poussart et al., 2004). Although microtomes are capable of extracting small, precise wood slivers (~10-µm thick), there are drawbacks to this method. First, because microtomes utilize a rotary blade, samples must be removed as parallel linear segments. Therefore wood samples must be prepared such that ring segments with straight-line or nearly straight-line boundaries can be sampled. Rings that are very narrow or rings that have tight curvatures, as is often the case in trees that grow under stress or are extremely

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doi:10.1016/j.chemgeo.2008.01.021

sensitive to environmental change, are difficult to sample without crosscutting growth rings, thereby mixing material from multiple time periods and thus reducing the resolution of the record. Second, it has been demonstrated that there is circumferential variability in $\delta^{18}O$, $\delta^{13}C$, and δD values due to morphological variations in leaves, stem cells, and bark at different positions around the tree. Although circumferential variation can be up to a few permil, these variations are not the result of environmental conditions and can distort the records of meteorological variables (e.g., Leavitt and Long, 1986). In order to obtain an average isotope value that represents the growth interval sampled, multiple sample transects would have to be prepared for sampling with a microtome.

Herein, we describe the use of a robotic micromilling device as an alternative high-resolution sampling method for obtaining subseasonal aliquots of cellulose concordant with growth rings. Robotic micromills are increasingly used for high-resolution sampling of gastropod and mollusc shells (e.g., Wurster and Patterson, 2001), fish otoliths (e.g., Patterson et al., 1993), animal teeth (e.g., Zazzo et al., 2006), and speleothems (e.g., Lachniet et al., 2004); however, to our knowledge, this is the first time it has been applied to isotope dendrochronology. Subseasonal variability observed in the intra-ring $\delta^{18}O$, $\delta^{13}C$, and δD values of α-cellulose indicate that high-resolution sampling of tree-ring cellulose offers the potential to reconstruct variation in meteorological conditions (e.g., temperature and precipitation) throughout the growing season.

2. Experimental section

2.1. Site descriptions and sample preparation

In order to compare methods for recovery of high-resolution cellulose samples with a robotic micromill, trees were sampled using a 1.2-cm Haglof™ increment borer and by cutting disks from felled trees. Samples discussed herein include white spruce (*Picea glauca*) and tamarack (*Larix laricina*) from central Saskatchewan, Canada as well as a poplar (*Populus grandidentata*) from Green Lakes State Park, Fayetteville, New York, USA. The tamarack and white spruce samples were collected in the Boreal Plain forest of central Saskatchewan, Canada. Trees in this forest ecosystem grow slowly due to the short growing season and low rainfall. Ring widths range from 0.1 mm to 4 mm in the white spruce, and from 1 mm to 3 mm in the tamarack (*L. laricina*). The poplar sample from Green Lakes State Park, New York, displayed greater variability in ring widths that ranged from 0.1 mm to >10 mm. The poplar sample was also felled by a storm in 1998 and therefore a complete disk of this sample was available. Core samples were trimmed into 10- to 15-cm lengths by slicing along ring boundaries with a scalpel, whereas disks were cut with a band saw into blocks approximately 15×5×2 cm (length×width×height). Samples were sanded with 100- to 500-grit sand paper to facilitate ring identification and prevent sampled powder from becoming caught in pores and cracks. Specimens were subsequently glued to glass dissection slides and attached to a moveable stage for micromilling.

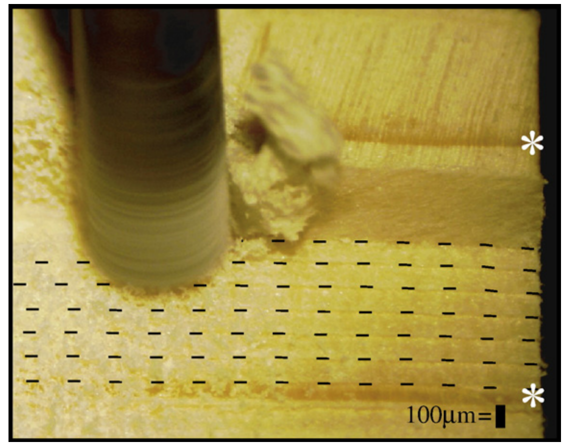

Fig. 1. Screen capture during the milling of a white spruce (1.2 cm diameter) core from central Saskatchewan. The growth ring boundaries (marked by white asterisks) are easily distinguished as well as previous sample paths (dashed lines). By discretely sampling with only the edge of the drill, samples that are much smaller than the diameter of the drill are collected. In this image, sample paths are 100 µm wide and the drill is moving left to right parallel to the ring boundaries.

2.2. Micromilling apparatus

Wood sections are fixed to a movable stage beneath a micro drill. Stage motion is controlled by three linear actuators with a step resolution of 0.05 µm on the *x*- and *y*-axes, and 1 µm on the *z*-axis. The apparatus is manipulated by a computer via a motion controller with a range of *x*-, *y*-, and *z*-axis motion of 17.78×7.62×4.54 cm, respectively. The micromilling device used in this study has been custom designed and was described in detail by Wurster et al. (1999).

Tree-rings are mapped using a color digital camera mounted on a fourth linear actuator. Digital characterization of tree-rings entails touching the tip of the drill to the surface of the sample at several points along the growth ring and recording these *x*–*y*–*z* coordinates. A cubic spline interpolation between the digitized points is subsequently generated that mimics three-dimensional circumferential variations in ring width. By characterizing two successive growth rings, multiple intermediate paths can be interpolated (Fig. 1). Spacing of intermediate paths can be regulated to insure that adequate material is recovered to generate sufficient gas for analyses. Due to the removal of up to 70% of wood material (lignin, resins, and other non-cellulose compounds) during α-cellulose processing, we determined that a minimum of 600 µg of raw wood is necessary to analyze δD, $\delta^{13}C$ and $\delta^{18}O$ from the same sample powder. For increment core samples, this generally requires a minimum path width of 400 µm; however, for the slabs where longer sample paths could be milled, individual path widths of <100 µm are possible.

Samples were milled using coarse cut carbide burs from Advanced Carbide Tool Company™. The total bur diameter (2.4 mm) is significantly greater than sample path widths. However, recovery of much smaller increments is made possible by milling (rather than drilling) concordant with ring boundaries such that only the edge of the bit comes in contact with the sample (Fig. 2). Milling depth is dependent upon the