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# Setting a Chronology for the Basal Ice at Dye-3 and GRIP: Implications for the Long-Term Stability of the Greenland Ice Sheet

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**Key Words:** Greenland Ice Sheet; Dye-3; GRIP; Ar-dating; <sup>17</sup>Δ stratigraphy

22 **Abstract**

23           The long-term stability of the Greenland Ice Sheet (GIS) is an important issue in our  
24 understanding of the climate system. Limited data suggest that the northern and southern sections  
25 extend well back into the Pleistocene, but most age constraints do not definitively date the ice.  
26 Here, we re-examine the GRIP and Dye-3 ice cores to provide direct ice core observations as to  
27 whether the GIS survived previous interglacials known to be warmer (~130 ka) or longer (~430  
28 ka) than that of the present interglacial. We present geochemical analyses of the basal ice from  
29 Dye-3 (1991-2035 m) and GRIP (3020-3026 m) that characterize and date the ice. We analyzed  
30 the elemental and isotopic composition of O<sub>2</sub>, N<sub>2</sub>, and Ar, of trapped air in these two cores to  
31 assess the origin of trapped gases in silty ice. Dating of the trapped air was then achieved by  
32 measuring the paleoatmospheric  $\delta^{40}\text{Ar}/^{38}\text{Ar}$  and the <sup>17</sup>O anomaly (<sup>17</sup>Δ) of O<sub>2</sub>. The resulting age is  
33 a lower limit because the trapped air maybe contaminated with crustal radiogenic <sup>40</sup>Ar. The  
34 oldest average age of replicates measured at various depths is 970 ± 140 ka for the GRIP ice core  
35 and 400 ka ± 170 ka for Dye-3. <sup>17</sup>Δ data from Dye-3 also argue strongly that basal ice in this core  
36 predates the Eemian. This confirms that the Greenland Ice Sheet did not completely melt at  
37 Southern Greenland during the last interglacial, nor did it completely melt at Summit Greenland  
38 during the unusually long interglacial ~ 430 kyr before present.

39

40 **Key Words:** Greenland Ice Sheet; Dye-3; GRIP; Ar-dating; <sup>17</sup>Δ stratigraphy

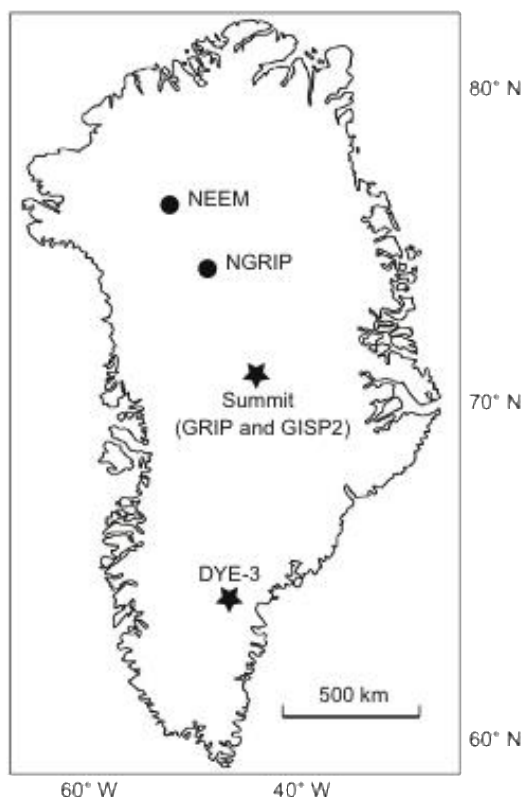
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## 42 **1. Introduction**

43           The Greenland Ice Sheet is the second largest reservoir of water on land, and if  
44 completely melted, would contribute roughly 7 m to global eustatic sea level (Bamber et al.,  
45 2013). As a large, climate sensitive source to sea level rise, the response of the Greenland Ice  
46 Sheet (GIS) to global warming has been an important, debated subject. Understanding the  
47 response of the GIS to past climate change events, such as previous interglacials, can provide a  
48 more accurate context for how anthropogenic global warming may impact the GIS in the future.  
49 Here, we advance the understanding of the long-term stability of the ice sheet by dating trapped  
50 air from the base of two Greenland ice cores (Fig. 1). By dating the trapped air from the deepest  
51 ice of the GRIP ice core, located at Summit Greenland, and the Dye-3 ice core, located in  
52 Southern Greenland, we determine, from direct observations, the minimum age after which an  
53 ice sheet existed at these sites. In establishing this age, we determine whether the GIS has  
54 persisted through interglacials such as the Eemian, when Arctic temperatures were 3-5°C warmer  
55 than today and sea level was 6-9 m higher than today (Clark and Huybers, 2009; Kopp et al.,  
56 2009) or Marine Isotope Stage 11 (400-430 ka), which was much longer than the present  
57 interglacial.

58           Previous studies on the trapped air in ice cores have established the antiquity of an ice  
59 sheet at Summit (GRIP and GISP2) and Northern Greenland (NEEM) through at least the last  
60 interglacial (115-130 ka), and probably back to Marine Isotope Stage 7, ~235 ka (Chappellaz et  
61 al., 1997; Suwa et al., 2006; Dahl-Jensen, 2013; Yau, PhD Thesis). However, an ongoing  
62 discussion persists on the minimum age of the deepest ice at Summit Greenland (GRIP) and  
63 Southern Greenland (Dye-3) because basal ice at these sites is stratigraphically disturbed  
64 (Johnsen et al., 2001; Verbeke et al., 2002; Tison et al., 1994; Bender et al., 2010). Of particular

65 interest is the question of whether the GIS survived at these sites through Marine Isotope Stage  
66 (MIS) 11, an unusually long interglacial when mean global sea level may have been 6-13 m  
67 above present (Raymo and Mitrovica, 2012).



68  
69 Fig. 1. Greenland ice core sites with possible Eemian-aged basal ice. Ice cores discussed in this  
70 study are starred.

71  
72 Ice at the base of the glacier has properties that originate in several different ways. “Dry  
73 densified ice” forms via the accumulation and burial of dry snow (Herron and Langway, 1980).  
74 “Wet origin ice” was partly or completely melted at some point in its history. Wet origin ice  
75 would include frozen soils, lake water, partly melted basal ice, and superimposed ice.

76 Dry densified ice has a total gas content of ~ 100 scc/kg (standard cubic centimeters of  
77 gas/kg of ice). The exact value depends on elevation (higher at low elevation), temperature

78 (higher at colder temperatures), and summer insolation. Wet origin ice has a much lower gas  
79 content (except for metabolic CO<sub>2</sub> and CH<sub>4</sub>), because the solubility of gas in water is much less  
80 than 100 scc/kg, and because gases may be exsolved upon freezing. If one mixes comparable  
81 amounts of dry densified and wet origin ice, the dry component will dominate the gas mixture.  
82 “Silty ice” sampled at the base of deep ice cores is generally dry origin ice mixed with some  
83 amount of either locally formed ice from the initial growth stages of the ice sheet (Tison et al.,  
84 1994; Souchez et al., 1994) or another basal component such as soil, permafrost, preglacial snow,  
85 lake ice, or ground ice (Bender et al., 2010). We use the term “clean ice” to describe ice free of  
86 visible silicate impurities. All clean ice samples in this paper are believed to be dry densified.

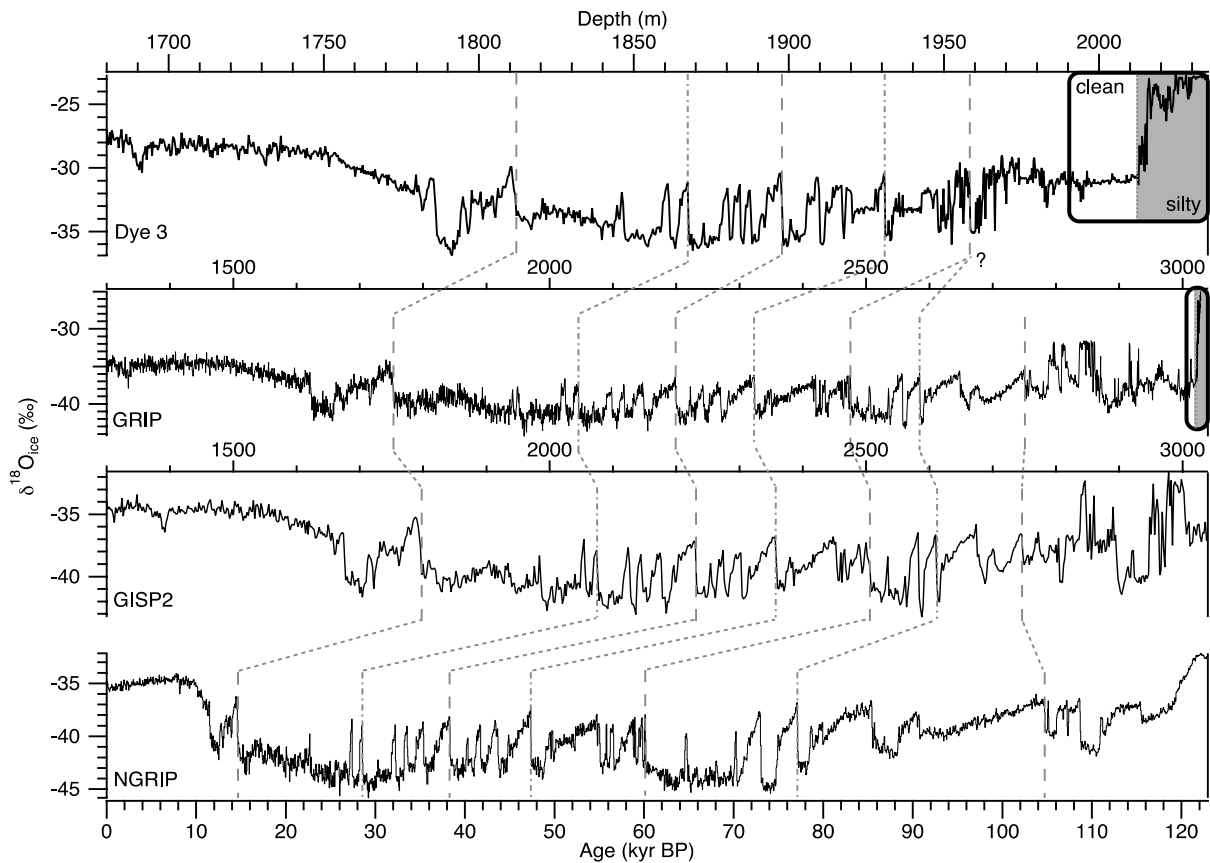
87         Previous studies have estimated that the silty basal ice, from 3022-3029 m depth in the  
88 GRIP ice core, could be as old as 2.4 Ma, dating to the original build-up of the GIS (Souchez et  
89 al., 1994; Souchez et al., 2006). Souchez et al. (2006) and Tison et al. (1998) found several  
90 indications of biological activity that indicated a soil source for the silty particles found in the  
91 bottom 7 m of GRIP. These included organic matter that was present in swampy areas, drawing  
92 down the O<sub>2</sub> concentration to 10% of saturation, very high concentrations of methane, high  
93 concentrations of NH<sub>4</sub><sup>+</sup>, and the presence of ammonium oxalate, which they attributed to the  
94 breakdown of bird droppings in local soils. They deduced that central Greenland was vegetated  
95 at the time the silty ice formed, and that traces of ancient soil were preserved in a snowdrift that  
96 was overridden by the ice sheet. <sup>10</sup>Be/<sup>36</sup>Cl dating by Willerslev et al. (2007) gives an age of the  
97 silty basal ice of 950 ± 44 ka. The uncertainty is calculated from <sup>36</sup>Cl and <sup>10</sup>Be abundance errors  
98 only, and the authors have noted several factors that could cause the <sup>36</sup>Cl/<sup>10</sup>Be age to be either  
99 younger or older than the true age of the ice. Here, we revisit the basal ice of the GRIP ice core

100 to confirm the antiquity of the trapped air of this ice inferred by Souchez et al. (1994, 2006) and  
101 Willerslev et al. (2007).

102 Willerslev et al. (2007) also concluded that basal ice from Southern Greenland at Dye-3  
103 is likely 450-800 ka. They presented results from a number of dating techniques (amino acid  
104 racemization,  $^{10}\text{Be}/^{36}\text{Cl}$ , and optical stimulated luminescence dating), each with its own set of  
105 assumptions and uncertainties, which collectively suggest that the Dye-3 silty ice dates to this  
106 age. However, the assumptions and uncertainties were large enough that Willerslev et al. (2007)  
107 were unable to conclusively rule out the possibility that the basal silty ice of Dye-3 may date  
108 only to the last interglacial (115-130 ka). Independent support for a pre-Eemian age for the  
109 southern GIS comes from sediment flux data from the southern Greenland margin (Colville et al.,  
110 2011; Reyes et al., 2014). While these studies have pointed to the antiquity and stability of the  
111 southern GIS through Arctic warm periods, earlier studies on the Dye-3 ice core inferred that the  
112 southern GIS did not persist through the Eemian. Koerner et al. (2002) argued that this region  
113 was ice-free during the last interglacial, as no isotopically cold ice exists below the warm basal  
114 section (Fig. 2). Other research highlights the abrupt and large increase with depth in  $\delta^{18}\text{O}_{\text{ice}}$   
115 ( $\sim 8\text{‰}$ ) over a very short interval (2012-2016 m). Koerner (1989) and Souchez et al., (1998)  
116 suggest that the silty basal ice is a completely separate unit of ice that was accreted onto the  
117 bottom of the overlying dry origin clean ice that makes up the bulk of the glacier at Dye 3 and  
118 GRIP, respectively (Fig. 2).

119 We analyzed samples from the clean and silty units of the Dye-3 ice core between 1991-  
120 2035 m and the GRIP ice core between 3020-3026 m. At Dye 3,  $\delta^{18}\text{O}$  of the ice is nearly  
121 constant between 1990-2012 m depth at  $-31\text{‰}$  (Holocene  $\delta^{18}\text{O} = -27\text{‰}$ ) (Fig. 2). Silty ice first  
122 appears at  $\sim 2012$  m in the Dye-3 ice core. From there to the bottom, at 2025 m depth,  $\delta^{18}\text{O}$  lies in

123 the range -24 to -26 ‰. At GRIP, silty ice first appears at 3022.25 m. From there to the bottom,  
 124  $\delta^{18}\text{O}$  rises from about -36 to -25‰. By focusing on the trapped air in ice, we mainly characterize  
 125 the dry densified ice component of the silty ice, rather than the wet origin component, which  
 126 bears the silt, and has a lower total gas content (Boereboom et al., 2013). However, wet based ice,  
 127 or other locally derived ice such as ice wedges, may still have substantial total gas contents  
 128 (Boereboom et al, 2013). Therefore, gas ages may be aliased by wet-origin ice predating the  
 129 growth of the ice sheet. Methods dating basal ice by measuring non-gas properties, such as U-  
 130 series recoil dating, primarily access the wet-origin ice, and may very well give different ages.



131  
 132 Fig. 2. Comparison of  $\delta^{18}\text{O}_{\text{ice}}$  for Dye-3, GRIP, GISP2, and NGRIP ice cores. Dye-3, GRIP, and  
 133 GISP2 are plotted on the top axis versus depth. NGRIP is plotted on the bottom axis versus age.  
 134 Dotted lines show  $\delta^{18}\text{O}_{\text{ice}}$  matching between cores. GRIP and GISP2 are chronologically



135 continuous to ~105 ka, and Dye-3 is chronologically continuous to ~60 ka. The bold boxes  
136 highlight the analyzed sections of Dye-3 and GRIP. The shaded portion indicates silty basal ice.

137

138 To determine the conditions during the trapping of air, we measured the elemental and  
139 isotopic composition of O<sub>2</sub>, N<sub>2</sub>, and Ar. We report the composition units with respect to air in the  
140 standard  $\delta$  notation with units in ‰ (per mil):

$$141 \quad \delta = [R/R_o - 1]$$

142 where  $\delta$  is the fractional deviation of a gas pair ratio  $R$  from a reference (air) ratio  $R_o$ . These  
143 analyses tell us whether the trapped air has been gravitationally or thermally fractionated ( $\delta^{15}\text{N}$   
144 and  $\delta^{38}\text{Ar}/^{36}\text{Ar}$ ), partially melted ( $\delta\text{Ar}/\text{N}_2$ ), or microbially respired ( $\delta^{18}\text{O}$  of O<sub>2</sub>,  $\delta\text{O}_2/\text{N}_2$ , and  
145  $\delta\text{O}_2/\text{Ar}$ ).

146 We also date the trapped air from both cores by measuring the paleoatmospheric  
147  $\delta^{40}\text{Ar}/^{38}\text{Ar}$  (Bender et al., 2008), and we present measurements of  $^{17}\Delta$  of O<sub>2</sub> (Blunier et al., 2002;  
148 Blunier et al., 2012) that help constrain the age of basal Dye-3 ice. These analyses contribute to  
149 our understanding of the origin of the basal ice from Dye-3 and GRIP, and how old this ice may  
150 be. These results provide useful constraints on the long-term stability of the GIS and models  
151 predicting its evolution in response to global change.

152

## 153 **2. Methods**

154 Interpreting gas properties requires a correction for gravitational fractionation. This term  
155 refers to the fact that, in a diffusive environment, heavy gases and isotopes are progressively  
156 enriched with depth according to the barometric equation. The enrichment scales with mass  
157 difference (Craig et al., 1988). We measure gravitational enrichment from  $\delta^{15}\text{N}$  and  $\delta^{38}\text{Ar}/^{36}\text{Ar}$ .

158 Gases may also be biased from the atmospheric composition by thermal fractionation. The effect  
159 of thermal fractionation is discussed in section 3.2.

160

## 161 **2.1 Age Reconstruction – Ar-chronometer**

162 We have dated the trapped air of the deepest GRIP and Dye-3 ice by the Ar-isotope  
163 method (Bender et al., 2008). The chronometer makes use of the fact that  $^{36}\text{Ar}$  and  $^{38}\text{Ar}$  have  
164 been essentially constant throughout recent geologic time, but  $^{40}\text{Ar}$  has been slowly increasing in  
165 the atmosphere as a result of the decay of  $^{40}\text{K}$ .  $\delta^{40}\text{Ar}/^{38}\text{Ar}$  has been measured in ice cores to 800  
166 ka. Over this period, it has risen at a rate of  $0.066 \pm 0.007\text{‰}/\text{Ma}$ . We precisely measure the  
167  $\delta^{40}\text{Ar}/^{38}\text{Ar}$  of the trapped air and determine the age of the air based on this rate of  $\delta^{40}\text{Ar}/^{38}\text{Ar}$   
168 increase.

169 The analytical method is similar to that described by Bender et al. (2008). Trapped air  
170 was extracted using a wet-melting technique, where ice was melted in an evacuated glass flask.  
171 The sample was then equilibrated with meltwater, and the meltwater was removed as outlined by  
172 Emerson et al. (1995). The sample gas was then passed through a water trap submerged in liquid  
173  $\text{N}_2$ , in which residual water and  $\text{CO}_2$  were frozen out. Ar was purified from the remaining gases  
174 through exposure to a getter which, when activated, reacts with and removes non-noble gases.  
175 The remaining gas was captured in a stainless steel tube submerged in liquid helium. After  
176 warming to room temperature, the sample was then analyzed on a Finnigan MAT 252 mass  
177 spectrometer with collectors specific to  $^{36}\text{Ar}$ ,  $^{38}\text{Ar}$ , and  $^{40}\text{Ar}$ .

178 Samples and air standards (modern air collected from the roof of the Princeton University  
179 Geosciences building in New Jersey) were run in three different analytical periods. The standard  
180 deviations of samples run in period 1 (n=7) for  $\delta^{40}\text{Ar}/^{38}\text{Ar}$ ,  $\delta^{38}\text{Ar}/^{36}\text{Ar}$ , and  $\delta^{40}\text{Ar}/^{36}\text{Ar}$  were

181  $\pm 0.018\%$ ,  $\pm 0.052\%$ , and  $\pm 0.035\%$  respectively. For period 2 (n=23), standard deviations were  
182  $\pm 0.016\%$ ,  $\pm 0.034\%$ , and  $\pm 0.020\%$ . For period 3 (n=7), standard deviations were  $\pm 0.008\%$ ,  
183  $\pm 0.023\%$ , and  $\pm 0.019\%$ . Table 1 indicates in which suite each sample was analyzed.  
184

**Table 1. Dating by Ar- and O<sub>2</sub>-isotope composition w.r.t. air. Each line represents a single analysis.**

GRIP ice core						
Depth (m)	Analysis Period <sup>o</sup>	$\delta^{40}\text{Ar}/^{38}\text{Ar}$ (‰)	$\delta^{40}\text{Ar}/^{36}\text{Ar}$ (‰)	$\delta^{38}\text{Ar}/^{36}\text{Ar}$ (‰)	$\delta^{40}\text{Ar}/^{38}\text{Ar}_{\text{atm}}$ (‰)	Age (ka)
3020	3	0.746	1.540	0.793	-0.048	710
3020	3	0.762	1.550	0.788	-0.027	390
3022	3	0.675	1.43	0.757	-0.083	1240
3022	3	0.713	1.482	0.77	-0.058	860
3025	3	0.454	0.963	0.509	-0.056	830
3025	-	-	-	-	-	-
3026	3	0.44	0.88	0.44	0	-10
3026	3	0.428	0.896	0.467	-0.039	580

\*Shaded section denotes samples from the silty basal ice

<sup>o</sup>See Methods

Analytical uncertainty for a single sample is  $\pm 250$  ka

Dye-3 ice core							
Depth (m)	Analysis Period <sup>o</sup>	$\delta^{40}\text{Ar}/^{38}\text{Ar}$ (‰)	$\delta^{40}\text{Ar}/^{36}\text{Ar}$ (‰)	$\delta^{38}\text{Ar}/^{36}\text{Ar}$ (‰)	$\delta^{40}\text{Ar}/^{38}\text{Ar}_{\text{atm}}$ (‰)	Age (ka)	$^{17}\Delta$ of O <sub>2</sub> (per meg)
2004	-	-	-	-	-	-	40.4
2004	-	-	-	-	-	-	29.6
2008	-	-	-	-	-	-	49.1
2008	-	-	-	-	-	-	47.2
2011	1	0.569	1.155	0.586	-0.017	260	-
2011	2	0.617	1.268	0.650	-0.035	530	-
2015	2	0.223	0.436	0.213	0.010	-150	-
2015	-	-	-	-	-	-	-
2019	1	0.238	0.462	0.224	0.014	-210	-
2019	2	0.150	0.285	0.135	0.015	-230	-
2023	-	-	-	-	-	-	36.1
2023	-	-	-	-	-	-	42.6
2031	2	0.196	0.382	0.186	0.010	-150	53.6
2031	-	-	-	-	-	-	48.0
2035	2	0.246	0.473	0.227	0.019	-280	-
2035	-	-	-	-	-	-	-

\*Shaded section denotes samples from the silty basal ice

<sup>o</sup>See Methods

Analytical uncertainty for a single sample is  $\pm 250$  ka

186 The term of merit for dating,  $\delta^{40}\text{Ar}/^{38}\text{Ar}_{\text{atm}}$  (paleoatmospheric  $\delta^{40}\text{Ar}/^{38}\text{Ar}$ ), is defined as

187 
$$\delta^{40}\text{Ar}/^{38}\text{Ar}_{\text{atm}} = \delta^{40}\text{Ar}/^{38}\text{Ar} - 1.002 * \delta^{38}\text{Ar}/^{36}\text{Ar} \quad (1)$$

188 The second term corrects for gravitational fractionation. The standard deviation of  $\delta^{40}\text{Ar}/^{38}\text{Ar}_{\text{atm}}$   
189 values, normalized to the means of their analysis periods, is  $\pm 0.016\text{‰}$  ( $n = 37$ ). The  
190 corresponding age uncertainty is  $\pm 250$  kyr ( $1\sigma$ ) for a single sample. During the first two analysis  
191 periods, 12 Holocene-aged ice samples (Newall Glacier ice core) were also analyzed using the  
192 same extraction technique applied to samples (one Newall sample was removed as an outlier)  
193 yielding an average age of  $130 \pm 150$  ka ( $1\sigma$ ). During the third analysis period, 3 samples  
194 inferred to have Holocene air (shallow ice from Mullins Valley, Antarctica with modern air due  
195 to cracks in the surface ice,  $< 5\text{m}$  depth) were analyzed, yielding an average age of  $110 \pm 230$  ka  
196 ( $1\sigma$ ).

197

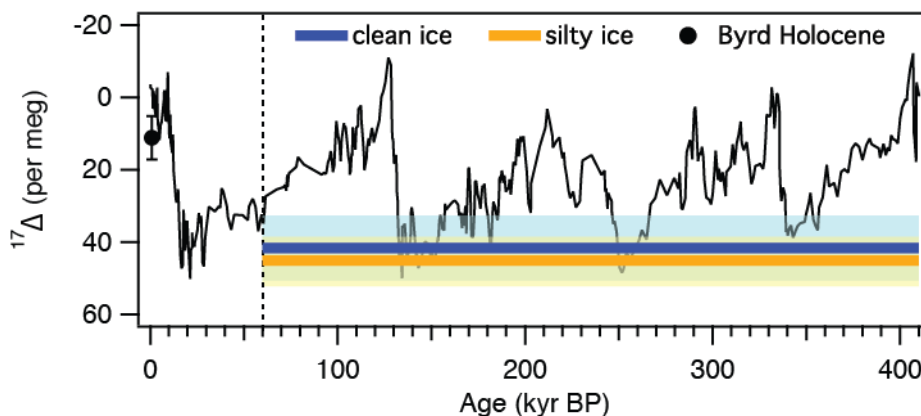
## 198 **2.2 Age Reconstruction – $^{17}\Delta$ of $\text{O}_2$ Stratigraphy**

199 A second technique was used to constrain the age of the basal ice at Dye-3, which  
200 involved analyzing the  $^{17}\Delta$  of  $\text{O}_2$  (the triple isotopic composition of  $\text{O}_2$ ), defined as:

201 
$$^{17}\Delta = (\ln(\delta^{17}\text{O}_{\text{atm}} + 1) - 0.516 * \ln(\delta^{18}\text{O}_{\text{atm}} + 1))$$
  
202 
$$\approx \delta^{17}\text{O}_{\text{atm}} - 0.516 * \delta^{18}\text{O}_{\text{atm}} \quad (2)$$

203 The coefficient (0.516) is the fractionation ratio for  $^{17}\text{O}/^{16}\text{O}$  of  $\text{O}_2$  relative to  $^{18}\text{O}/^{16}\text{O}$  (Luz et al.,  
204 1999). This technique exploits the fact that  $^{17}\Delta$  is a measure of the non mass-dependent  
205 fractionation of  $\text{O}_2$ , which originates in  $\text{O}_2$ - $\text{CO}_2$  exchange in the stratosphere (Luz et al., 1999).  
206  $^{17}\Delta$  changes with time in the atmosphere, and the record is known for the past 400 ka from the  
207 Vostok, Siple, and GISP2 ice cores (Fig. 3; Blunier et al., 2012). To a first approximation,  $^{17}\Delta$

208 tracks contemporaneous CO<sub>2</sub> (Blunier et al., 2012). <sup>17</sup>Δ data are given in per meg where 1 per  
209 meg corresponds to 0.001 ‰.



210  
211 Fig. 3. Mean of <sup>17</sup>Δ of O<sub>2</sub> for Dye-3 clean and silty ice plotted versus age. In black, a 3-point  
212 smoothing of <sup>17</sup>Δ of O<sub>2</sub> for the past 400 ka from Vostok, GISP2, and Siple (Blunier et al., 2012).  
213 In yellow, <sup>17</sup>Δ of O<sub>2</sub> for silty ice; mean = 45 ± 7 per meg (1σ in light yellow; st. error = ±4 per  
214 meg). In blue, <sup>17</sup>Δ of O<sub>2</sub> for clean ice; mean = 42 ± 9 per meg (1σ in light blue; st. error = ±4 per  
215 meg). The black circle is the average of Byrd Holocene samples; mean 11 ± 6 per meg (1σ). The  
216 dashed line marks 60 ka, the minimum age for the deep ice (see Fig. 2). Mean values for both the  
217 clean and silty ice are comparable to glacial maximum values observed for deep ice cores,  
218 indicating the air is at least as old as 132 ka.

219  
220 Given the definition of <sup>17</sup>Δ, microbial respiration of air (a mass-dependent fractionation  
221 process) does not contaminate this record, as it causes δ<sup>17</sup>O<sub>atm</sub> to change at a rate equal to 0.516  
222 times the change in δ<sup>18</sup>O<sub>atm</sub>. In contrast, metabolic production or consumption of gases in silty  
223 ice alters all other properties used for gas stratigraphy in ice cores, including CO<sub>2</sub>, CH<sub>4</sub>, and  
224 δ<sup>18</sup>O<sub>atm</sub> (paleoatmospheric δ<sup>18</sup>O of O<sub>2</sub>). This is an important distinction as the basal silty ice of  
225 Dye-3 is contaminated by microbial respiration, with CO<sub>2</sub> values observed up to 36,000 ppmv

226 (Souchez et al., 1998); consequently, these gas properties cannot be used for chemostratigraphy.  
227 Here, we make use of the fact that saw-tooth, 100-kyr glacial-interglacial CO<sub>2</sub> cycles are  
228 observed in the <sup>17</sup>Δ record (Blunier et al., 2002; Blunier et al., 2012), which can be used to place  
229 samples in the context of the global climate state.

230 The triple isotope composition of oxygen (<sup>17</sup>Δ) was determined by processing air  
231 extracted from ~50 g of ice through a vacuum line connected to a gas chromatograph as per  
232 Blunier et al. (2002). <sup>17</sup>Δ is calculated by using δ<sup>15</sup>N to gravitationally correct both δ<sup>17</sup>O and  
233 δ<sup>18</sup>O, which are then used in eqn. (2). The analytical uncertainty in <sup>17</sup>Δ, based on the analysis of  
234 modern air standards, is ±6 per meg (1σ, n = 6). 5 Holocene-aged ice samples (Byrd ice core  
235 ~200-500m depth) were also analyzed using the same sampling technique applied to ice core  
236 samples, yielding a Holocene <sup>17</sup>Δ of 11 ± 6 per meg (1σ). All <sup>17</sup>Δ values are normalized to that of  
237 modern air.

238

### 239 **2.3 Analyses of the Elemental and Isotopic Composition of O<sub>2</sub>, N<sub>2</sub>, and Ar**

240 Measurements of the elemental and isotopic composition of air (δ<sup>15</sup>N, δ<sup>18</sup>O of O<sub>2</sub>, δAr/N<sub>2</sub>,  
241 δO<sub>2</sub>/N<sub>2</sub>, and δO<sub>2</sub>/Ar) were performed using the same wet-melting extraction technique applied  
242 for the Ar-dating. In these extractions, ~20g of ice were used, and samples were passed through a  
243 water trap in liquid nitrogen and directly captured in a 12 scc stainless steel tube submerged in  
244 liquid helium. The sample was then warmed to room temperature and analyzed on a Finnigan  
245 Delta Plus XP mass spectrometer. The standard deviation of modern air standards (n = 8),  
246 processed as samples, were: δ<sup>15</sup>N = ±0.016‰; δ<sup>18</sup>O of O<sub>2</sub> = ±0.023‰; δAr/N<sub>2</sub> = ±0.24‰;  
247 δO<sub>2</sub>/N<sub>2</sub> = ±0.45‰; δO<sub>2</sub>/Ar = ±0.34‰. The paleoatmospheric δ<sup>18</sup>O of O<sub>2</sub>, δ<sup>18</sup>O<sub>atm</sub>, is equal to

248  $\delta^{18}\text{O}$  corrected for gravitational fractionation:  $\delta^{18}\text{O}_{\text{atm}} = \delta^{18}\text{O} - 2.01 * \delta^{15}\text{N}$ . The standard  
249 deviation is  $\pm 0.024\text{‰}$ .

250

### 251 **3. Results and Discussion**

#### 252 **3.1 Characteristics of dry densified ice**

253 Typical dry densified ice has about 100 scc of air per kg (Herron and Langway, 1987).

254 Holocene ice at Summit has a total gas content of  $\sim 90$  scc/kg, and the total gas content of  
255 Holocene Dye-3 ice is likely higher (Martinerie et al., 1992).

256 Dry densified ice is also characterized by near-zero values of  $\delta\text{Ar}/\text{N}_2$ ,  $\delta\text{O}_2/\text{N}_2$ , and  $\delta\text{O}_2/\text{Ar}$   
257 taking ambient air as the reference (Sowers et al., 1989; Bender et al., 1995). Values for  $\delta\text{Ar}/\text{N}_2$   
258 typically range between  $-10\text{‰}$  and  $0\text{‰}$  in dry densified ice. These ratios are slightly depleted due  
259 to the preferential loss of Ar relative to  $\text{N}_2$  during bubble close-off, because Ar atoms are smaller  
260 than  $\text{N}_2$  molecules (Craig et al., 1988).  $\delta\text{Ar}/\text{N}_2$  values above  $0\text{‰}$  are indicative of partially  
261 melted and refrozen ice, as the solubility ratio of  $\text{Ar}/\text{N}_2$  is approximately 2/1.  $\delta\text{O}_2/\text{N}_2$  and  $\delta\text{O}_2/\text{Ar}$   
262 values of dry densified ice are generally slightly lower than the ratios in air, and are between -  
263  $25\text{‰}$  to  $0\text{‰}$  (Sowers et al., 1989; Bender et al., 1995). Depletions below these ratios indicate  
264 microbial respiration, which results in the consumption of  $\text{O}_2$  (Souchez, 1997). Microbial  
265 respiration also results in the enrichment of  $\delta^{18}\text{O}_{\text{atm}}$  (modern air =  $0\text{‰}$ ; paleo-atmospheric range  
266 =  $-0.4$  to  $+1.4$  (Dreyfus et al., 2007)), as microbes preferentially consume  $^{16}\text{O}$  relative to  $^{18}\text{O}$ .

267 In the firn densification process, air is fractionated by gravity, which results in the  
268 enrichment of heavier isotopes with depth (Craig et al., 1988; Schwander, 1989). Gravitational  
269 fractionation occurs in the firn layer and is preserved in ice once the close-off density is reached.  
270  $^{15}\text{N}$  is enriched in the trapped air of dry origin ice, with  $\delta^{15}\text{N}$  typically between  $0.2$ - $0.5\text{‰}$



271 (modern air = 0‰), corresponding to a close-off depth of ~40-100 m. This means that for every  
272 20 m depth increase in the firn,  $\delta^{15}\text{N}$  is enriched by ~0.1‰. If the basal ice of GRIP and Dye-3 is  
273 formed by the dry compaction of snow in a climate similar to that of today and at the top of the  
274 central ice sheet, we would expect to find the following characteristics: total gas content ~100  
275 scc/kg;  $\delta^{15}\text{N}$  from ~-0.2-0.5‰; values of  $\delta^{18}\text{O}_{\text{atm}}$  within the paleo-atmospheric range (about -  
276 0.4‰ to +1.5‰); and Ar/N<sub>2</sub> values slightly below that of modern air.

277         The presence of liquid water will force elemental ratios of  $\delta\text{Ar}/\text{N}_2$ ,  $\delta\text{O}_2/\text{N}_2$ , and  $\delta\text{O}_2/\text{Ar}$   
278 towards water saturation ratios rather than atmospheric ratios in wet origin ice (Fig. 4). A buried  
279 perennial snowbank may have elemental ratios close to that of air, and its trapped air would be  
280 gravitationally fractionated. However, the magnitude would be small because of the limited  
281 depth, and might be attenuated by convection (Severinghaus et al., 2010). Snowbank air could be  
282 thermally fractionated as well. (In thermal fractionation, temperature gradients associated with  
283 seasonal temperature fluctuations can fractionate atmospheric ratios and isotopes both negatively  
284 and positively; Chapman and Cowling, 1970; Severinghaus et al., 2001.) Low total air contents,  
285 elemental ratios drawn towards water saturation ratios, and variable isotopic signatures  
286 (reflecting contributions of different processes) would be expected in wet origin basal ice.

287

### 288 **3.2 Characteristics of GRIP and Dye-3 Basal Ice**

289         Table 2 and Figs. 4 and 5 summarize the geochemical data for the trapped air of the deep  
290 GRIP and Dye-3 ice.  $\delta\text{Ar}/\text{N}_2$  is plotted vs.  $\delta\text{O}_2/\text{Ar}$  for the GRIP and Dye-3 samples, along with  
291 data for various examples of wet origin ice (Fig. 4; Cardyn et al., 2007; Lacelle et al., 2011).

292         Air trapped in firn from Dye-3 and GRIP have the following characteristics. First, Ar/N<sub>2</sub>  
293 ratios are close to atmospheric (Fig. 4). Ratios are slightly elevated in silty GRIP (+37‰) and

294 Dye-3 (+26‰) ice, perhaps due to a small wet origin component (Souchez et al., 1988; Knight,  
295 1997). However, they are nowhere near the saturation value (~+1000‰). Second, N<sub>2</sub> and Ar  
296 isotopes both show normal gravitational enrichments (Fig. 5). The exceptions are in the two  
297 deepest dirty GRIP samples, where  $\delta^{38}\text{Ar}/^{36}\text{Ar}$  is much less than  $2 \times \delta^{15}\text{N}$ , and in the silty Dye-3  
298 ice, where the enrichments are only ~0.1‰ per mass unit. Third, total air content values in silty  
299 ice from GRIP and Dye-3 are significantly lower than the overlying clean ice (Table 3; Souchez  
300 et al., 1995a; Souchez et al., 1998). And fourth, evidence for microbial respiration is clear with  
301  $\delta^{18}\text{O}_{\text{atm}}$  most enriched in samples that are most depleted in O<sub>2</sub> based on the  $\delta\text{O}_2/\text{Ar}$  value (Fig. 5).  
302 GRIP silty ice is characterized by unusually large consumption of O<sub>2</sub> and very enriched  $\delta^{18}\text{O}_{\text{atm}}$   
303 values. These observations are all consistent with CO<sub>2</sub> and CH<sub>4</sub> concentrations, and O<sub>2</sub>  $\delta^{18}\text{O}$   
304 values observed by Souchez et al. (1995b; 2006). The most parsimonious explanation for these  
305 observations is that basal ice at GRIP and Dye-3 is composed primarily of dry densified clean ice,  
306 with some contribution of silty, wet origin ice. A quantification of mixing components is not  
307 attempted here, but has been described in other studies (Souchez et al., 1995a; Souchez et al.,  
308 1998; Verbeke et al., 2002; Bender et al., 2010).

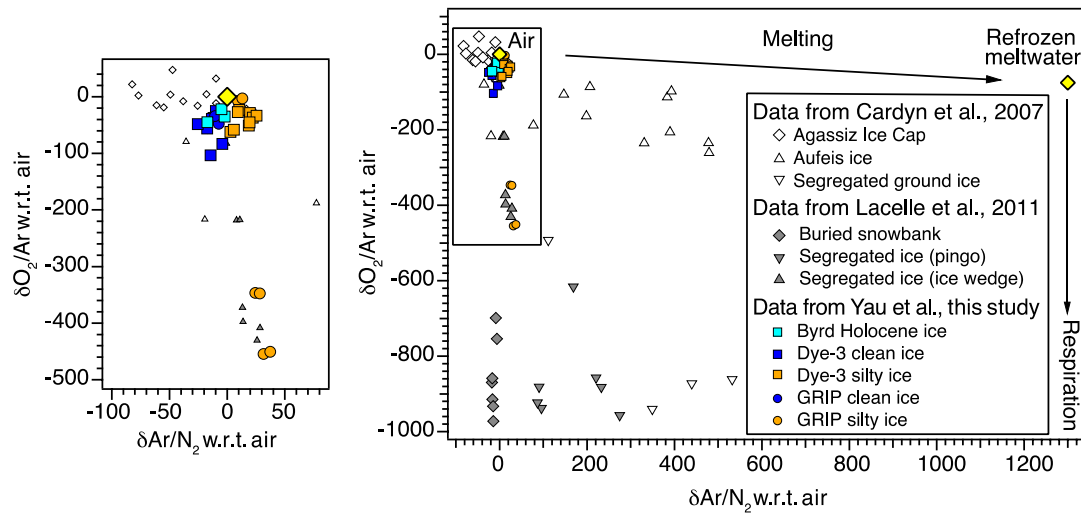
309

### 310 **3.3 Age Constraints on the Basal Ice of GRIP and Dye-3**

311 Dating of the trapped air reflects the average age of the dry densified glacial ice and  
312 locally formed ice, with the components weighted according to their abundance and total gas  
313 content. Based on the relatively high total gas content, the local ice component is likely to play a  
314 small role in the Ar isotope ages of the most important samples in this study (GRIP at 3022 m  
315 and Dye-3 at 2011 m depth). Table 1 summarizes the measured Ar-isotope ages for the deepest  
316 ice at GRIP and Dye-3. For GRIP, 4 samples from 3020-3026 m were analyzed. The deepest

317 sample replicated poorly, with one replicate dating to the present. The anomalously young age of  
318 this replicate may reflect incorporation of radiogenic  $^{40}\text{Ar}$  from outgassing of the local  
319 underlying crust, as seen in basal silty ice at GISP2 (Bender et al., 2010). Consequently, we  
320 exclude this sample from our discussion of the age of the GRIP basal ice, and infer that the Ar-  
321 ages from the basal ice section are lower limits because the trapped air may be contaminated  
322 with crustal radiogenic  $^{40}\text{Ar}$ . It is possible that wet origin ice could cause Ar isotope ages to be  
323 older than the age of the dry densified ice. This influence would only be important if the age of  
324 the trapped air from the wet origin ice was not reset by contamination associated with  
325 contraction cracks (St. Jean et al., 2011), before it was overridden by dry densified glacial ice.  
326 The clean GRIP sample from 3020 m depth dates to  $550 \pm 170$  ka ( $n=2$ ; 1 st. error), and the  
327 remaining silty samples from 3022 m and 3025 m date to  $970 \pm 140$  ka ( $n=3$ ; 1 st. error). The  
328  $^{40}\text{Ar}$  ages for the silty basal ice agree with the  $^{10}\text{Be}/^{36}\text{Cl}$  age of Willerslev et al. (2007),  $950 \pm 44$   
329 ka. We therefore conclude that the silty basal ice from GRIP certainly dates to well before MIS  
330 11 (430 ka). It is possible that the GRIP silty basal ice dates to the original build-up of the GIS,  
331 as originally proposed by Souchez et al. (1994).  
332

333



334

335 Fig. 4. Right:  $\delta\text{Ar}/\text{N}_2$  vs.  $\delta\text{O}_2/\text{Ar}$  for deep GRIP and Dye-3 ice compared with Holocene ice from

336 Byrd, Antarctica and Agassiz Ice Cap, Canada, and various deposits of wet origin ice. Left:

337 Zoom-in on GRIP and Dye-3 ice. The deepest Dye-3 and GRIP ice have  $\delta\text{Ar}/\text{N}_2$  values similar to

338 that of typical dry densified ice. Some small enrichments in  $\delta\text{Ar}/\text{N}_2$  may be due to regelation of

339 ice or some small mixing with a wet origin ice component.  $\delta\text{O}_2/\text{N}_2$  of the silty GRIP ice indicate

340 significant respiration.

341

**Table 2. Gas composition w.r.t. modern air. Each line represents a single analysis.**

GRIP ice core						
Depth (m)	$\delta\text{O}_2/\text{N}_2$ (‰)	$\delta\text{O}_2/\text{Ar}$ (‰)	$\delta\text{Ar}/\text{N}_2$ (‰)	$\delta^{15}\text{N}$ (‰)	$\delta^{18}\text{O}_{\text{atm}}$ (‰)	$\delta^{18}\text{O}_{\text{ice}}$ (‰)
3020	-54.65	-47.79	-7.38	0.34	0.87	-
3020	-43.85	-33.88	-10.51	0.37	0.69	-
3022	2.18	-7.37	9.42	0.27	0.38	-34.4
3022	10.36	-2.88	13.08	0.33	0.34	-34.4
3025	-330.55	-346.50	24.30	0.46	4.87	-28.3
3025	-328.92	-347.53	28.35	0.46	4.79	-28.3
3026	-437.32	-454.58	31.51	0.50	5.91	-28.0
3026	-430.15	-450.77	37.32	0.53	5.76	-28.0

\*Shaded section denotes samples from the silty basal ice

Dye-3 ice core						
Depth (m)	$\delta\text{O}_2/\text{N}_2$ (‰)	$\delta\text{O}_2/\text{Ar}$ (‰)	$\delta\text{Ar}/\text{N}_2$ (‰)	$\delta^{15}\text{N}$ (‰)	$\delta^{18}\text{O}_{\text{atm}}$ (‰)	$\delta^{18}\text{O}_{\text{ice}}$ (‰)
1991	-48.75	-37.45	-11.92	0.28	0.36	-31.1
1991	-72.38	-56.12	-17.40	0.29	0.54	-31.1
1999	-116.25	-103.53	-14.38	0.35	1.50	-31.2
1999	-87.25	-83.50	-4.29	0.25	1.25	-31.2
2004	-34.52	-25.04	-9.90	0.27	0.60	-31.0
2004	-72.29	-48.20	-25.49	0.33	0.75	-31.0
2008	-47.31	-37.29	-10.40	0.33	0.51	-31.1
2008	-51.60	-39.06	-13.23	0.30	0.65	-31.1
2011	-	-	-	-	-	-30.6
2011	-	-	-	-	-	-30.6
2014	-11.95	-31.74	20.23	0.16	0.74	-27.9
2014	-9.21	-28.54	19.71	0.17	0.68	-27.9
2015	-	-	-	-	-	-26.6
2015	-	-	-	-	-	-26.6
2019	-	-	-	-	-	-24.0
2019	-	-	-	-	-	-24.0
2023	-13.04	-23.20	10.40	0.20	0.62	-24.5
2023	-17.96	-27.12	9.22	0.13	0.70	-24.5
2027	-15.41	-36.97	22.20	0.10	0.84	-23.4
2027	-8.57	-33.40	25.50	0.07	0.81	-23.4
2031	-32.96	-50.81	18.80	0.16	0.71	-22.9
2031	-26.43	-45.15	19.43	0.07	0.91	-22.9
2035	-59.20	-62.08	2.88	0.11	1.10	-22.8
2035	-52.69	-58.27	5.74	0.12	1.01	-22.8

\*Shaded section denotes samples from the silty basal ice

**Table 3. Total Air Content**

GRIP ice core		Dye-3 ice core	
Depth (m)	Air Content (scc/g)	Depth (m)	Air Content (scc/g)
3022.3	0.098	2010.9	0.081
3022.5	0.084	2012.5	0.089
3022.6	0.068	2014.1	0.069
3022.9	0.077	2014.5	0.089
3023.3	0.087	2015.3	0.090
3023.7	0.077	2016.1	0.053
3024.1	0.080	2016.7	0.067
3024.4	0.068	2018.3	0.061
3024.7	0.070	2021.7	0.068
3025.1	0.063	2022.7	0.060
3025.5	0.058	2023.3	0.075
3025.7	0.074	2024.7	0.053
3026.2	0.065	2025.3	0.065
3026.4	0.068	2027.1	0.056
3026.6	0.072	2027.5	0.057
3027.3	0.059	2028.7	0.064
3027.9	0.051	2029.1	0.053
3028.3	0.048	2029.7	0.053
3028.6	0.048	2031.1	0.052
		2032.1	0.055
		2032.9	0.059
		2033.9	0.047
		2034.7	0.059
		2035.5	0.059
		2035.9	0.052
		2036.7	0.062
		2037.4	0.051
		2037.8	0.036

\*Data for GRIP from Souchez et al. (1995a). Data for Dye-3 from Souchez et al. (1998). For Dye-3, Units 1-3, as defined by Souchez et al. (1998), are noted by shading.

344

345

For Dye-3, 1 sample was analyzed from the clean section, and 4 from the silty section;

346

one sample was replicated from each section. The average age for the silty basal ice is  $210 \pm 110$

347

kyr (1 st. error, n=2) in the future. This means that there is excess  $^{40}\text{Ar}$  in the basal ice at Dye-3

348

that is driving the  $\delta^{40}\text{Ar}/^{38}\text{Ar}_{\text{atm}}$  value above that of the modern atmosphere. The amount of

349

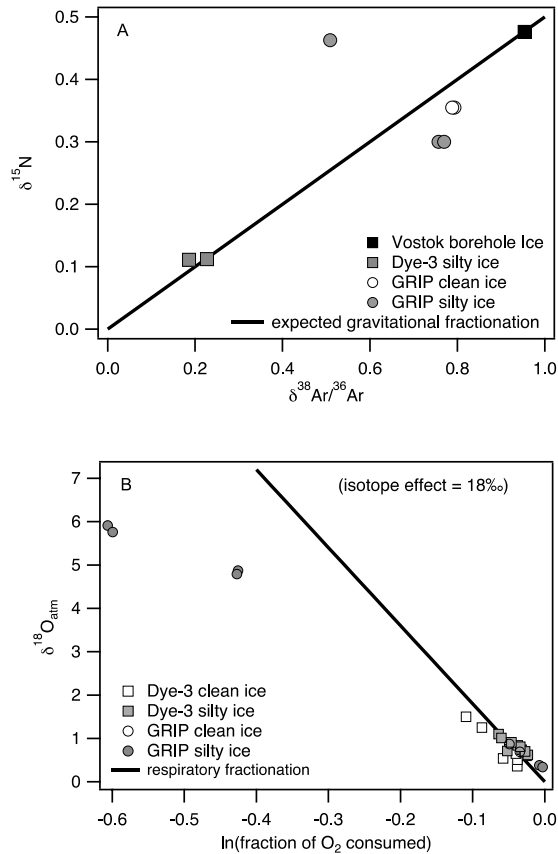
excess  $^{40}\text{Ar}$  far exceeds the amount that could be generated from radiogenic  $^{40}\text{Ar}$  produced by

350 just the silt and debris in the ice (weight % between 0.05-0.6%, Olmez et al., 1993). The likely  
351 source of excess  $^{40}\text{Ar}$  is outgassing of radiogenic  $^{40}\text{Ar}$  from the underlying continental crust, as is  
352 seen at GISP2 and GRIP (Bender et al., 2010; this work).

353 The Ar-isotope age for replicates from the clean ice (2011 m) is  $400 \pm 170$  ka (1 st. error,  
354  $n=2$ ). This result is fully consistent with data in Willerslev et al. (2007) suggesting an age of 400-  
355 800 ka for basal ice at Dye-3. However, the large Ar age uncertainty prevents a definitive  
356 conclusion at the 95% confidence level that Dye-3 was ice-covered during the Eemian.  
357 Consequently, analyses of  $^{17}\Delta$  of  $\text{O}_2$  are performed to further constrain the age.

358 For at least the past 400 ka,  $^{17}\Delta$  has varied systematically with climate and atmospheric  
359  $\text{CO}_2$  (Fig. 3). Interglacial  $^{17}\Delta$  typically averages -5 to +10 per meg, while glacial values average  
360 between +35 and +45 per meg (Blunier et al., 2002; Blunier et al., 2012). With an analytical  
361 uncertainty of  $\pm 6$  per meg,  $^{17}\Delta$  can date trapped air to glacial or interglacial times.

362 Following Oeschger et al. (1983), we believe that Dye-3 is basically stratigraphically  
363 intact back to about 60 ka (Fig. 2). In this view, the  $\delta^{18}\text{O}$  peak at 1932 m corresponds to  
364 Interstadial 12 in GISP2 and GRIP (~48 ka). The maximum at 1935 m corresponds to  
365 Interstadial 13, and the maximum at 1945 m corresponds to Interstadial 14. At 1956 m and 1958  
366 m in Dye-3, 2 isotope maxima correspond to Interstadial 15 and the short unnumbered  
367 interstadial that follows. Then at 1955 m and 1958 m depth, two particularly warm events  
368 separated by a cold event correspond to Interstadials 16 and 17 in GISP2 and GRIP. According  
369 to these correlations, Dye-3 has an age of 60 ka at 1959 m depth, and deeper ice is presumed to  
370 be older.



371  
 372 Fig. 5. A. Plot of  $\delta^{38}\text{Ar}/^{36}\text{Ar}$  vs.  $\delta^{15}\text{N}$ . The heavy black line is the expected mass-dependent  
 373 gravitational fractionation. A Vostok borehole (~100 m depth) sample illustrates how air from  
 374 typical ice is fractionated. The Dye-3 silty ice and GRIP clean ice are gravitationally fractionated,  
 375 though we observe scatter in the deepest silty GRIP samples. B. Plot of the fraction of  $\text{O}_2$   
 376 consumed (based on the deviation of  $\text{O}_2/\text{Ar}$  from the atmospheric ratio) vs.  $\delta^{18}\text{O}_{\text{atm}}$ . The heavy  
 377 black line is the Rayleigh fractionation relationship assuming a respiratory isotope effect of 18‰.  
 378 Samples from the deep Dye-3 (clean and silty) and GRIP (clean) ice plot along this line,  
 379 indicating that some  $\text{O}_2$  in the trapped air has been consumed by respiration. GRIP silty basal ice  
 380 samples show an unusually large degree of consumption. Our  $\delta^{18}\text{O}$  data are similar to earlier  
 381 results of Souchez et al. (2006).  
 382



383 With this constraint, we use  $^{17}\Delta$  to determine the youngest probable age of the trapped air.  
384 Fig. 3 shows the results of the  $^{17}\Delta$  analyses. 4 clean ice samples had  $^{17}\Delta$  values of  $+42 \pm 9$  per  
385 meg (1 $\sigma$ ; st. error =  $\pm 4$  per meg). 4 samples of silty ice had  $^{17}\Delta$  values of  $+45 \pm 7$  per meg (1 $\sigma$ ; st.  
386 error =  $\pm 4$  per meg). These are diagnostic of glacial maximum air and (inferentially) ice (Fig. 3).  
387 Given that the minimum age for the deep ice is 60 ka, and that prior to this time,  $^{17}\Delta$  values of  
388  $\sim 43$  per meg were not realized until  $\sim 132$  ka, we conclude that Dye-3 was ice-covered through  
389 the LIG. This interglacial was exceptionally warm. It thus seems likely that the GIS at Dye-3 was  
390 intact since the unusually long Marine Isotope Stage 11, and perhaps at earlier times.

391

#### 392 **4. Conclusions**

393 We have analyzed the concentration and isotopic composition of O<sub>2</sub>, N<sub>2</sub> and Ar in  
394 trapped gases from clean and silty basal ice from the GISP2 and DYE-3 ice cores. Ar/N<sub>2</sub> ratios,  
395 and  $\delta^{15}\text{N}$  of N<sub>2</sub>, confirm earlier work (including Souchez et al., 2006) suggesting that trapped  
396 gases in silty ice derive primarily from clean, dry densified ice in most samples.  $^{40}\text{Ar}/^{38}\text{Ar}$  ratios  
397 constrain ages with an uncertainty of 150-250 ka for a single sample. Contamination by crustal  
398 radiogenic  $^{40}\text{Ar}$  make ice ages minimum ages, and in Dye-3 the deepest samples have ages in the  
399 future. These limitations notwithstanding, our data have significant implications for the history  
400 of the Greenland Ice Sheet.

401 Analyses of the trapped air from the GRIP and Dye-3 basal ice indicate a relatively  
402 resilient GIS. Ar-isotope dating of air from the silty basal ice gives a minimum age for the ice  
403 sheet at Summit of  $970 \pm 140$  ka (1 st. error), suggesting that the GIS at Summit survived  
404 through MIS 11 despite significant collapse of the GIS (Raymo and Mitrovica, 2012). In addition,  
405 evidence that the ice sheet at Dye-3 survived the last interglacial comes from two Ar-isotope

406 dates averaging  $400 \pm 170$  ka (1 st. error).  $^{17}\Delta$  stratigraphy further constrains the minimum age to  
407  $\sim 132$  ka. Ice dating to the last interglacial was not found in the depth interval studied (1991-2035  
408 m), and does not appear to be present at shallower depths, where  $\delta^{18}\text{O}_{\text{ice}}$  values do not reach  
409 Holocene values ( $\sim -28\%$ ; Johnsen et al., 2001). We presume that ice dynamics removed Eemian  
410 ice from the Dye-3 site, but the record holds no information about how this might have happened.

411       It remains a matter of investigation whether the ice sheet at Dye-3 was a part of the main  
412 body of the GIS, or whether an ice dome at Southern Greenland was present during the Eemian  
413 (Cuffey and Marshall, 2000; Huybrechts, 2002; Tarasov and Peltier, 2003; Lhomme et al., 2005).  
414 Our results do not preclude extensive melting of the ice sheet over the LIG, as changes at the  
415 margin as well as in coastal northern Greenland could have been extensive (Born and  
416 Nisancioglu, 2012), and the net elevation change at Dye-3 during the LIG is unknown.

417       Our data indicate that the GIS did not completely melt at Southern Greenland during the  
418 Eemian, nor did it completely melt at Summit Greenland during MIS 11. These constraints on  
419 the trapped air of basal ice from GRIP and Dye-3 are in line with estimates of the age and  
420 stability of the GIS from Willerslev et al. (2007), Colville et al. (2011), and Reyes et al. (2014).

421

422

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428

429

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