A FIB-NanoSIMS-TEM study of Unmelted Antarctic Micrometeorite TT006D107. M. Ito<sup>1</sup>, N. Tomioka<sup>1</sup>, Y. Kodama<sup>2</sup> and N. Imae<sup>3</sup>. <sup>1</sup>Kochi Institute for Core Sample Research, JAMSTEC (B200 Monobe, Nankoku City, Kochi Japan 783–8502, motoo@jamstec.go.jp). <sup>2</sup>Marine Works Japan. <sup>3</sup>National Institute of Polar Research.

**Introduction:** Micrometeorites are dominant flux of the extraterrestrial materials for the accreting of the Earth [1]. So far several types of micrometeorites (Antarctic micrometeorites: AMMs) were collected from Antarctic snow and ice [e.g., 2, 3]. AMM is classified into (1) unmelted type (fine-grained, coarse-grained), (2) partially melted scoriaceous type and (3) melted type based on their texture. Chemical and mineralogical data indicate that most of the AMMs are related to carbonaceous chondrites (CI, CM, and CR types) [4]. In this study, we report a systematic isotopic and mineralogical investigations for unmelted Antarctic micrometeorite by using NanoSIMS ion microprobe and TEM/STEM to constrain its nature and origin.

Experiments: We used an unmelted micrometeorite, TT006D107, as a sample that collected at Tottuki point in Antarctica in 2000 [3]. The TT006D107 is irregularly shaped with  $\sim 60 \times 50 \mu m$  in size, and was pressed onto the Gecko Tape made by carbon-nanotube (Nitto Denko corp.). A first investigation by an EPMA with EDS (JEOL JXA-8200) was carried out at National Institute of Polar Research to obtain bulk elemental abundances, seconday election and back-scattered electron images of the sample. The sample was, then, sent to the JAMSTEC Kochi Institute of Core Sample Research for further investigation. A cross-section of the sample  $(30 \times 30 \times 2 \mu m)$  was prepared by the single FIB (Hitachi SMI4050) (Fig. 1a). We have applied an ion imaging by the JAMSTEC NanoSIMS 50L to acquire isotope images of H, C, N and O as well as elemental images of Si, Mg as MgO, Al as AlO, Ca as CaO, and Fe as FeO of the sample (Table 1 for trolley settings). All images were acquired by rastering a 1-3pA 16keV  $Cs^+$  primary ion beam over area of 25 µm x 25 µm. Each image run repeatedly scanned over (20-40 times) the same area, with individual images consisting of 256 x 256 pixels, having a dwell time of 5,000 µs/pixel. The spatial resolution was estimated to be ~100 nm for O, C and N isotope images (the first and second settings) and  $\sim$ 200 nm for H isotpe image (the third setting). The sample was transferred into the FE-SEM (JEOL JSM-6500F at Kochi University) for observation of the sample surface. We, then, determined the detailed mineralogy and microstructure of the same area that we acquired isotope/elemental images to gain insight into its petrogenesis by TEM (JEOL ARM-200F equipped with EDS) followed by the single FIB to prepare an ultra thin section for TEM.

Table 1.	Trollev	settings	for each	n measurement

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Detectors	1 <sup>st</sup> setting	2 <sup>nd</sup> setting	3 <sup>rd</sup> setting		
#1	160	12C	1H		
#2	18O	13C	2D		
#3	28Si	160			
#4	24Mg16O				
#5	27Al16O	12C14N			
#6	40Ca16O	12C15N			
#7	54Fe16O	32S	12C		

**Results:** The  $\delta^{18}$ O isotopic composition in mineral phases of the sample shows homogeneous distribution of +18 ± 2 permil (Fig. 1b). Engrand and Dbrica [5] found the O isotopic varitations in unmelted (-5 to +15.5 permil in  $\delta^{18}$ O) and partially melted (+6 to +32 permil in  $\delta^{18}$ O) AMMs. Matrajt and coworkers reported systematic investigations of O isotopic compositions (+3 to +60 permil in  $\delta^{18}$ O) in 28 unmelted AMMs [6]. The O isotopic composition of the TT006D107 unmelted AMM is consistent with that of previous AMMs [e.g., 5-7].

We obtained isotope images of  $\delta D$ ,  $\delta^{13}C$  and  $\delta^{15}N$ for carbonaceous materials (C and N-rich regions) in the sample (Fig. 1c-e). The carbonaceous materials in the sample show bulk values of  $\delta D$ ,  $\delta^{13}C$  and  $\delta^{15}N$  of +2,424 ± 62 permil, -2 ± 5 permil and +50 ± 8 permil, respectively. The bulk  $\delta D$  values is good agreement with values from CR, OC or CM (Bells) chondrites [8, 9]. The bulk  $\delta^{13}C$  and  $\delta^{15}N$  are similar to the those of CI and CM (Bells or heated) chondrites [9], and  $\delta^{15}N$  is good agreement with other AMMs [10]

We found a nano globule with a size of ~ 200–300 nm $\phi$  in the measured area (Fig. 1c-e). Its isotopic compositions were  $\delta D = +1,450 \pm 300$  permil,  $\delta^{13}C = +44 \pm 22$  permil and  $\delta^{15}N = +900 \pm 77$  permil that are similar to the values that reported in previous study [e.g., 11]. It is noted that  $\delta D$  value of the nano globule is smaller than that value of bulk  $\delta D$  in the sample.

We have conducted STEM/TEM observations for part of the sample. The unmelted AMM consists mainly of Mg-Fe-Al-bearing amorphous silicate. Fe-Ni-S-O spherules smaller than < 20 nm in diameter were found to be embedded in the amorphous material, although their mineral phases could not be identified due to their complex electron diffraction patterns.

**Discussions:** This enriched  $\delta^{18}$ O (+18 permil) of the AMM could be the effect of atmospheric entry heating owing to the O isotopic compositions of partially melted and unmelted AMMs [5]. In contrast, Matrajt et al. [6] reported that the O isotopic compositions preserved the

original values, and did not change during the atmospheric entry. However, we are not able to distinguish both scenarios proposed in [5, 6] in terms of O isotopic composition.

Isotopic compositions and microstructural observations of the sample play a critical role in understaning of the origin and nature of unmelted AMMs. Matrajt et al. [6] proposed that the AMMs might be originated from a CI-like chondrite parent body due to distributions of their O isotopic compositions. The bulk  $\delta^{13}$ C and  $\delta^{15}$ N suggested that the origin of the AMM may be CI and CM (Bells or heated) chondrites [8]. Texture observation by TEM suggested that the AMM experienced moderate heating because of the exsistence of SiO<sub>2</sub>-rich amorphous with Fe-O particles. In previous reports [12, 13], phyllosilicates (e.g., Fe-rich serpentine) in CM and CI chondrites transformed to SiO<sub>2</sub>-rich amorphous material with Fe-O particles under moderate heating and dehydration processes. These isotopic and microtextural characteristics imply that the origin of the AMM could be CI or CM-like parent body, and the AMM have experienced moderate heating during either atmospheric entry and/or thermal process in the parent body.

UCAMM is a carbonaceous materials-rich micrometeorite having extreamly high D/H ratio collected from Antarctic blue ice [e.g., 14]. The similarities between UCAMM and the AMM are 1) heterogeneous distribution of D having a high D/H isotopic ratio, and 2) moderate to high C/H ratio in C-rich region of the sample (0.1 to 2.5 for the AMM, 0.4 to 8.5 for the UCAMM) [14]. A relationship between unmelted AMMs and UCAMM may provide an important clue to the early Solar System organics together with its associated minerals.

Further investigations of individual unmelted AMMs are essential to constrain on their origin and nature, and the connection to UCAMMs, IDPs, IOMs in chondrites and cometary returned samples by STARDUST mission.

**References:** [1] Love S.G. and Brownlee D.E. (1993) Science 262, 550-553. [2] Maurette M. et al. (1986) Science 233, 869-872. [3] Iwata N. and Imae N. (2002) Antarct. Meteorite Res. 15, 25-27. [4] Kurat G. et al. (1994) GCA 58, 3879-3904. [5] Engrand C. and Dobrica E. (2012) 43rd LPSC abstract#2636. [6] Matrajt G. et al. (2006) GCA 70, 4007-4018. [7] Engrand C. et al. (1999) GCA 63, 2623-2636. [8] Alexander C.M.O'D. et al. (2007) GCA 71, 4380-4403. [9] Busemann H. (2006) Science 312, 728-730. [10] Haenecour P. et al. (2014) 11th Int. GeoRaman Conf. abstract#5017. [11] Nakamura-Messenger K. et al. (2006) Science 314, 1439-1441. [12] Toppani et al. (2001) MAPS 36, 1377-1396. [13] Tomioka N. et al (2007) MAPS 42, 19-30. [14] Duprat J. et al. (2010) Science 328, 742-745.

