


( ※本報告書は英語で記述してください。ただし、産業利用課題として採択されている方は日本語で記述していただいても結構です。 )

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課題番号 Project No. 2012A0048  実験課題名 Title of experiment Evaluation of Aggregation and Agglomeration of nano-materials with the small angle neutron scattering technique  実験責任者名 Name of principal investigator Takeshi Eitoku  所属 Affiliation Advanced Institute of Science and Technology	装置責任者 Name of responsible person Junichi Suzuki  装置名 Name of Instrument/(BL No.) BL-15 TAIKAN  実施日時 Date and time of Experiment 2 <sup>nd</sup> June, 2012

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。(適宜、図表添付のこと)  
Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.  Four different forms of Carbon nanotube (CNT) dispersed solution (D <sub>2</sub> O) with 0.1% polymer Triton X-100 Detail of CNT forms are; (1) Thick and hard rod (30 nm x 1 um) multiwall CNT, made by ark-discharge method as grown. (2) Same as (1) but purified samples. (3) Tangled like cotton (10nm x 0.2-0.3 um) multiwall CNT, made by CVD method as grown (4) Same as (3) but purified samples
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2. 実験方法及び結果 (実験がうまくいかなかった場合、その理由を記述してください。) Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.  Method; two different forms of MWNT(multiwall carbon nanotube) dispersion, and blank solution (0.1% Triton-X100, D2O solution) were measured by SANS technique. Based on our previous TEM measurement, the size and forms were as follows; (1)(2) Hard and straight rod-like; the diameter is around 30-40 nm and the length is about 1 um This hard, straight shape is consistent with the Raman G/D ratio measurement, which shows the smaller amount of defect is included in this MWNT. (1) Since the “as grown” samples include a little amount of impurities, MWNT tends to connect each other and form a larger particle. (2) In the case of “purified” sample, in which the impurities graphite is removed, major component is a mono-dispersed rod, and large particle and bundle is a minor.
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## 2. 実験方法及び結果(つづき) Experimental method and results (continued)

(3)(4) Very flexible MWNT, the diameter is around 10nm and the length is about 0.3  $\mu\text{m}$  or so. Since this MWNT is not straight, the evaluation of length is difficult and inaccurate. Also this MWNT tends to form a large size of tangled particle by interconnecting each other. The existence of impurities may cause a crucial effect for the formation of large tangled shape, which is not clear.

Detailed measurement conditions are as follows;

Sample (1), 1mg/ml, exposure time; 1h

Sample (2), 1 mg/ml, exposure time; 3h ( see result figure (left))

Sample (2), 0.1 mg/ml, exposure time; 3h

Sample (3), 1mg/ml, exposure time; 1h

Sample (4), 1 mg/ml, exposure time; 3h ( see result figure (right) )

Sample (4), 0.1 mg/ml, exposure time; 3h

Blank solution sample; 3h

Neutron intensity monitor (no sample) ; 3h

Scattered neutron of  $\lambda = 0.5\text{--}7.8 \text{ \AA}$  was detected with time-resolved device and distinguished by TOF of neutron, and analyzed by  $0.1 \text{ \AA}$  step collection.

Result; Subtle difference could be found between the scattering patterns of MWNT samples and blank solutions as shown in the following figures, in the  $q$ -range of  $0.02\text{--}0.1 \text{ \AA}^{-1}$ , which correspond to several nanometers in real-space. The structural information of single CNT and high-order CNT-CNT interaction were very difficult to be extracted from these SANS result. If the detectors at smaller angles are installed and the  $q$ -range of TAIKAN is expanded to the lower  $q$  region in the near future, we will be able to obtain the various information about the form and structure about single CNT and CNT-CNT interactions, and clarify the nature about the aggregation and agglomeration of CNT dispersion solution. Thus, the SANS measurement using TAIKAN will have a significant importance and great potential in the field of material science in the near future.

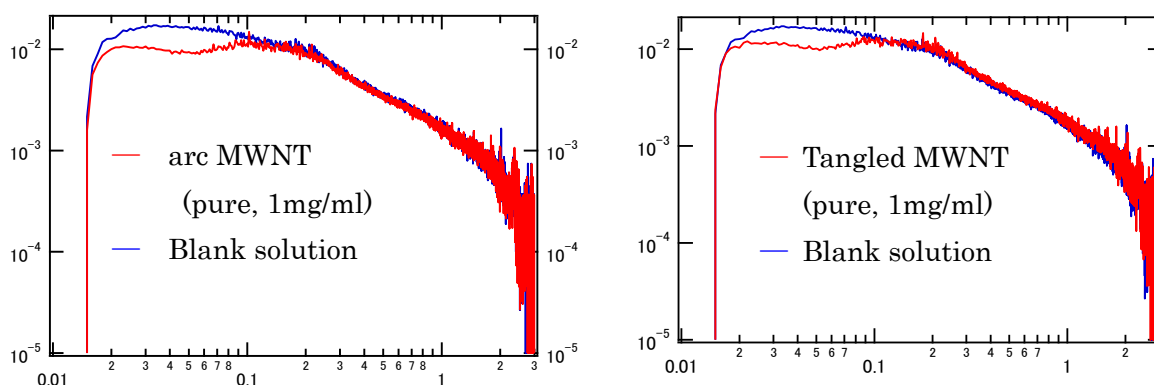


Figure : SANS profile of Multiwall Carbon nanotube dispersion and blank solutions. (Left) MWNT made by Arc method (Right) Tangled MWNT by CVD