# FORMATION OF TUNNEL BARRIERS AND QUANTUM DOT STRUCTURE IN SUSPENDED CARBON NANOTUBES BY FOCUSED ION BEAM FOR SINGLE ELECTRON TRANSISTOR

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### **DEDICATION**

This thesis is dedicated to my mother, who taught me that the best kind of knowledge to have is that which is learned for its own sake. It is also dedicated to my family, who taught me that even the largest task can be accomplished if it is done one step at a time.

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#### ABSTRAK

Karbon tiub nano (CNT) mewakili kelas baru bagi peranti nano berdasarkan titik kuantum (QD) kerana diameternya sekecil 10nm tidak dapat dibina oleh teknik piawaian litografi. Sifat mekaniknya yang unik seperti jisim yang ringan amat sesuai bagi penyalun yang berfrekuensi tinggi dengan keupayaan pengesanan ultrasensitif. Kajian ini menunjukkan satu proses yang boleh digunakan bagi membina penghalang terowong di karbon tiub nano multi-dinding yang terampai (MWCNTs) dengan menggunakan teknik pemindahan mekanikal untuk pertama kalinya. Teknik ini juga dapat membuka jalan untuk membina peranti berfungsi mekanikal berasaskan tiub nano. Rintangan dua terminal di setiap peranti diukur sebelum pembentukan penghalang terowong oleh sinar ion terfokus (FIB) iaitu sekitar  $10k\Omega$  hingga  $20k\Omega$ . MWCNT yang terampai kemudiannya diimbas menggunakan FIB. Sebanyak 5 sampel dibina untuk setiap dos ion yang sama, iaitu dari  $1.5 \times 10^{16}$  ions/cm<sup>2</sup> hingga 6× 10<sup>16</sup> ions/cm<sup>2</sup> dan kesan perubahan rintangan setelah penyinaran akan dikaji. Hasil kajian ini menunjukkan bahawa rintangan meningkat dengan peningkatan dos Ga ion untuk tiub nano pada substrat dan tiub nano yang terampai. Menariknya, bagi kes tiub nano yang terampai, dos yang lebih tinggi diperlukan untuk meningkatkan rintangan berbanding dengan tiub nano pada substrat. Data dianalisis pada suhu rendah serendah 1.5 K dan graf Arrhenius telah di plot. Terdapat kecenderungan bahawa ketinggian penghalang mempunyai hubungan dengan rintangan yang meningkat setelah penyinaran yang dikendalikan oleh dos pancaran ion dan diameter tiub nano. SET telah dibina dalam MWCNT terampai dengan membentuk dua halangan. Dalam kajian ini, transistor elekton tunggal (SET) yang dibina dari MWCNT yang terampai telah berjaya di hasilkan menggunakan teknik yang mudah dan jimat dimana berlian Coulob dan ayunan Coulomb dapat di ukur.

#### ABSTRACT

Carbon nanotube (CNT) represents a new class of building blocks for quantum do (QD) based nanodevices and circuits due to its extremely small diameter of 10nm whereby the standard lithography technique cannot easily realize such dimension. In addition to their electrical and optical applications, their unique mechanical properties such as light mass and large stiffness are attractive for a highfrequency resonator with a possible ultrasensitive mass sensing capability. This research demonstrates for the first time a reliable process to fabricate tunnel barriers in the suspended multi-wall carbon nanotubes (MWCNTs) by the mechanical transfer technique. This technique may also open a way to fabricate nanotube based mechanical functional devices. The two-terminal resistance of each device was measured before the formation of tunnel barriers ranging from  $10k\Omega$  to  $20k\Omega$  by focused ion beam (FIB). The suspended MWCNTs was then single scanned using FIB. More than 5 samples were fabricated for each ion dose from  $1.5 \times 10^{16}$  ions/cm<sup>2</sup> to  $6 \times 10^{16}$  ions/cm<sup>2</sup> and the effect of the resistance changed after irradiation was studied. This result showed that the resistance increased with rising Ga ion dose for both nanotubes on a substrate and the suspended nanotubes. Interestingly, for the case of the suspended nanotubes a higher dose was necessary to increase the resistance compared to the nanotube on a substrate. To analyse the data further, the sample was cooled down in a liquid helium refrigerator from room temperature to the lowest temperature of 1.5 K. The estimation of barrier height by Arrhenius plot obtained was then plotted as a function of resistance change after irradiation. There was a tendency that the barrier height correlated with the increased resistance after irradiation that was controlled by the dose of ion beam and diameter of the nanotubes. The single electron transistor (SET) was fabricated in suspended MWCNT by forming two barriers. The regular Coulomb diamonds and Coulomb oscillations were observed and some sample showed spike-like noise, superimposed on the regular Coulomb oscillations. It can be concluded that the SET was successfully fabricated in suspended MWCNT using simple and low-cost technique wherein Coulomb diamond and Coulomb oscillation were successfully observed.

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## LIST OF ABBREVIATIONS

-	Atomic Force Microscopy
-	Aluminium
-	Argon
-	Aurum
-	buffered oxide etch
-	Carbon Nanotubes
-	Chromium
-	Chemical Vapour Deposition
-	De-Ionized Water
-	Electron beam lithography
-	Focus Ion Beam
-	Frequency Modulation
-	Gallium
-	Hydrogen
-	Helium
-	Hydrofluoric
-	Isopropyl Alcohol
-	Current Voltage
-	Multi Wall Carbon Nanotubes
-	Nitrogen
-	Palladium
-	Polymethyl Methacrylate
-	Polyvinyl Alcohol
-	Resistance
-	reactive ion etching
-	Room Temperature
-	Scanning Electron Microscope
-	Single Electron Transistor
-	Silicon

SiC	-	Silicon Carbide
SIO	-	Silicon-on-insulator
SiO <sub>2</sub>	-	Silicon Dioxide
SWCNT	-	Single Wall Carbon Nanotubes
Ti	-	Titanium
UV	-	Ultraviolet
QD	-	Quantum Dots

## LIST OF SYMBOLS

°C	-	Degree Celsius
μ	-	Micro
n	-	Nano
e	-	Elementary Charge
С	-	Capacitance
Rt	-	Tunnel Resistance
Rq	-	Quantum Resistance
h	-	Plank Constant
k	-	Boltzmann constant
Т	-	Temperature
Ec	-	Coulomb energy
GHz	-	Giga Hertz
MHz	-	Mega Hertz
Ω	-	Ohms
С	-	Centi
А	-	Ampere
р	-	Pico
Å	-	Angstrom
K	-	Kelvin
σ	-	Conductivity
L	-	Length
A/d2	-	Area
φb	-	Barrier Height
Cth	-	Constant Thermally Excited Carriers
Ctu	-	Constant Tunnelling Process
m	-	mili
Vsd	-	Source Drain Voltage
М	-	Mega
Ec	-	Charging Energy

aF	-	Attofarad
Cg	-	Gate Capacitance
Vg	-	Gate Voltage
V	-	Volt

#### **CHAPTER 1**

#### **INTRODUCTION**

#### 1.1 Research background

The mechanical resonator is highly desirable in various applications to detect tiny amounts of mass in mass sensors [1-3], such as the high sensitivity detection of bacteria [4,5], monitoring air pollution [6,7], evaluating hydrogen storage capacity [8] and others. These applications have stimulated scientists to conduct intensive research to achieve the goals of scaling down the resonator and improving the measurement of devices. Since the principle of detecting vibration in the resonator is rather challenging, a new device structure and measurement technique should be developed. Several types of transducers - such as beam silicon carbide (SiC) [9-11], silicon nanowires [12,13] and carbon nanotubes (CNTs) [14,15] - have recently been explored to fabricate the mechanical resonator. However, SiC [16] and silicon nanowire [17] transducers cannot gain the high-resonance frequency that is crucial for the operation of devices.

The carbon nanotube (CNT) represents a new class of building blocks for quantum dot (QD)-based nanodevices and circuits [18-20] due to its extremely small diameter, whereas the standard lithography technique cannot easily realise such dimensions. Although the diameter of a multi-wall CNT (MWCNT) is slightly larger than that of a single-wall CNT (SWCNT), the MWCNT seems to be more favourable because of its robustness in conventional semiconductor processing technology. In addition, the capability of a metal/MWCNT contact to sustain its ohmic characteristics, even down to liquid He temperature [21], makes the MWCNT an ideal channel material for the fabrication of QD-based nanodevices and circuits. CNTs have also been used to fabricate mechanical resonators that can operate at ultra high frequencies [3]. In addition, CNTs can also show ballistic electron transport and a Coulomb blockade [9]. The coupling of the mechanical motion of the nanotube resonator and the electron transport will also offer an interesting functionality in devices. However, forming a tunnel barrier to function as a QD remains a difficult task. In previous studies, the fabrication of tunnel barriers in MWCNTs on a SiO<sub>2</sub>/Si substrate by Ar ion irradiation [22-24] was reported. However, Ga ion irradiation is more favourable because its resistance variation is far lower than that of Ar ion beam irradiation. Ga focused ion beam (FIB) was a useful technique for fabricating tunnel barriers in our structure. In previous work, researchers claimed that the Ga ion has lower resistance variation than the Ar ion, probably due to the experimental setup using Ar ion irradiation, which needs a wide resists opening to expose the substrate because of the limitation of photolithography equipment. Section 2.7 will discuss the factors that contribute to resistance variation for the Ar ion beam.

To fabricate mechanical vibration devices, the nanotube should be suspended; however, fabricating a suspended nanotube is still challenging. We have successfully demonstrated, for the first time, a reliable process of fabricating tunnel barriers in suspended multi-wall carbon nanotubes (MWCNT) by the mechanical transfer technique. This technique was modified from the transfer process commonly used to produce a van der Waals heterostructure using two-dimensional materials [25,26]. This technique seems to be a promising approach for fabricating a straight suspended CNT, compared to other techniques such as wet etching the underlying oxide layer below the nanotube using hydrofluoric acid [27]. The technique for fabricating a suspended nanotube is explained in detail in section 2.6.

The yield of the suspended nanotubes from wet etching is relatively low due to the collapse of the nanotube on the substrate. Tunnel barriers in MWCNTs will be formed using FIB, since this technique is far more reliable than Ar ion irradiation, especially in forming the precise location of tunnel barriers to create small QDs. This work presents and discusses the effects of control parameters such as irradiation dose and nanotube diameter on the tunnel barrier properties, namely nanotube resistance and barrier height, for suspended nanotubes. The basic characteristics of a fabricated single-electron transistor (SET) in a suspended nanotube are also presented.

### **1.2** Research motivation

Electromechanical resonators are widely employed to detect tiny amounts of mass, which can be used to monitor the deposition rates from an evaporation source, air pollution and so forth. Since the principle used in the resonator for detecting vibration is rather challenging, a new device structure and measurement technique should be developed. The CNT represents a new class of building blocks that offers a unique attribute: a small diameter, which cannot be realised by the standard lithography technique. Its properties of light mass and stiffness make it ideal for ultrasensitive force detection. In addition, a CNT can also show ballistic electron transport and a Coulomb blockade at room temperature [28]. The coupling of the mechanical motion of the nanotube resonator and the electron transport will also offer an interesting functionality in the relevant devices. In particular, we expect that this coupling can be used to control the mechanical motion of nanoscale schemes [29]. With the advantage of the CNT being that can it be fabricated as an SET, such a structure can be fully utilised to demonstrate an electromechanical resonator. In this work, a QD on the suspended CNT will be fabricated using the FIB technique. This SET structure is expected to be able to control the mechanical vibration when it is applied as a resonator. This device is expected to be a powerful high-frequency resonator tool that would conceal novel research into using the electromechanical resonator system in classical and quantum regimes.

#### **1.3** Originality of this work

Several research studies have been reported the fabrication of QDs in MWCNTs on a substrate. However, little research has reported the fabrication of QDs in suspended MWCNTs. Normally, researchers have fabricated the suspended nanotube using the wet etching technique. However, this technique has some disadvantages, whereby the yield production is relatively low because the nanotube collapses easily on the substrate.

In this work, we introduce a new technique for fabricating suspended MWCNTs, using the mechanical transfer approach for the first time. We found that

this technique is more reproducible compared to the wet etching technique using HF. The mechanical transfer technique used in this work was modified from the transfer process commonly used to produce a van der Waals heterostructure using two-dimensional materials.

It is still challenging to produce reproducible QDs on devices. In previous work, researchers successfully fabricated QDs on a substrate using Ar atom [30] and Ar ion [31] irradiation. However, they found that using Ar ion beam irradiation on the nanotube created too much damage, causing devices to break and fail. They also reported that the Ga ion beam is a promising technique for producing tunnel barriers in MWCNTs. However, they still observed some variation in the measured source-drain resistance, even though the same dose of ion beam irradiation was applied to the sample. They could not find the cause of the resistance variation, even though the same parameters were used in each experiment.

In this work, we studied the fabrication of an SET in suspended MWCNTs and systematically investigated the cause of the resistance variation by carefully analysing each parameter during SET fabrication. The resistance variation comes from either the diameter of the nanotubes or the ion dose. An on-chip CNT was also fabricated as a reference to investigate how much Ga ion dose must be applied to the nanotube during irradiation, because fabricating a suspended CNT is more complicated than an on-chip CNT. Therefore, with the reference sample of the nanotube on the chip, we could estimate how much ion dose can be applied to the suspended carbon nanotube. The mechanical transfer technique was used to fabricate gub in the MWCNTs. This will be a promising technique for fabricating a high-frequency mechanical resonator [32].

#### **1.4 Problem statement**

The development of a high-frequency mechanical resonator is promising for new applications, such as sensitive mass/gas sensors [1-3], biological imaging [33] and mechanical devices for high-frequency signals processing [34]. Since the principle used in the resonator, which is the detection of vibration, is rather challenging, a new device structure and measurement technique should be developed. In a previous report, researchers used an AFM cantilever [35] to detect the vibration of nanotubes when current was applied to the electrode. However, it was very difficult to detect the vibration and the device was not sensitive enough, especially when it was applied as a high-frequency resonator. For a device to function as a high-frequency resonator, the selection of materials is crucial.

As reported by Huang, a SiC [36] resonator was successfully fabricated with a resonance frequency that could reach 1 GHz. Mechanical resonators can also be fabricated using Si nanowires; however, the resonance frequency is only 94.7 MHz [37]. Other researchers used SiO [38] for making a mechanical resonator, but the frequency they measured was 5 MHz. We think that the MWCNT is suitable for fabricating a mechanical resonator because it has great mechanical strength and is a material with a light mass. In previous work, D. Garcia [35] reported the successful fabrication of an MWCNT resonator with a resonance frequency that could reach 3.1 GHz. However, in this report, the researchers just studied the mechanical properties of the devices, stating that the mechanical resonator's vibration was hard to detect. As mentioned before, since the detection aspect of a high-frequency mechanical detector is very challenging, a new device structure and measurement technique should be developed. The SET coupling to the mechanical resonator can be an alternative way to overcome this challenge [39,40].

According to the principle of fabricating the mechanical resonator, the nanotube should be suspended. However, it is still a challenging task to fabricate reproducible suspended nanotubes. There are several techniques for fabricating a suspended MWCNT. One is by making the trench using wet etching, normally with hydrofluoric acid, to etch the silicon under the nanotubes [41]. However, the nanotube easily lay down on the Si substrate when the trench was made using this technique. This would make the nanotube channel impossible to use as the resonator. Another technique for fabricating a suspended nanotube is to use the resists technique and tilt the metal deposition to make the nanotube become suspended [42]. However, nanotubes fabricated by the resists technique also laid down on the substrate after deposition. Therefore, in this work, we introduced a new technique for systematically fabricating a reproducible suspended MWCNT, using the mechanical transfer technique for the first time. The mechanical transfer technique is far more reproducible because it simply picks any nanotube using an optical microscope and transfers it to any desired location. The nanotube was properly suspended when checked under an SEM.

After fabricating the suspended MWCNT, we will create a barrier on the MWCNT. We need at least two barriers to form the QD, and the region between the two barriers will work as the QD. Various techniques can be used to fabricate tunnel barriers. One of these is the resists opening technique using Ar ion beam irradiation [31]. However, this technique is not reproducible, so the resistance value varies greatly from one sample to another, even though the same parameters might be applied for each sample. This is probably due to the low controllability of the 50 nm resists opening area or the resists resolution, which makes the Ar ion beam hit the nanotube inconsistently. As a result, the resistance varies considerably from one sample to another. To overcome the resists resolution problem, we used FIB Ga ion beam irradiation to fabricate the tunnel barriers to obtain a closer and more precise location.

#### **1.5** Research objective

The objectives of this study are as follows:

i) To develop a reliable technique for fabricating suspended CNTs, using the mechanical transfer technique for the first time.

ii) To demonstrate a reliable process of fabricating tunnel barriers in the suspended MWCNTs by Ga focused ion beam and to systematically study the effect of resistance changes during tunnel barrier fabrication.

iii) To show that fabricated devices with suspended MWCNTs can be used as SETs.

#### **1.6** Research scope

This work involved the fabrication of the suspended MWCNTs, using the mechanical transfer technique for the first time. After successfully fabricating suspended MWCNTs using this technique, we studied a reliable process of fabricating tunnel barriers using Ga focused ion beam irradiation and investigated the cause of the resistance changes in the sample. In the process of fabricating the tunnel barriers, the size and ion dose of the nanotubes were important parameters. These were systematically studied to analyse the variations in resistance changes after ion beam irradiation. We also studied the relationship between the barrier height, the diameter of the nanotubes and the dose of Ga ion beam applied on the nanotubes in creating damage or tunnel barriers. The fabricated tunnel barrier in each suspended CNT was tested to determine whether it could be used as an SET. To observe the behaviour of the SET, we analysed its current-voltage characteristics and the conductance of the fabricated samples at cryogenic temperature. Finally, we demonstrated the SET and studied the possibility that it could be used as a resonator. The research on the mechanical resonator referred to other previously reported work. The current work mainly focused on experimental work to fabricate an SET in suspended CNTs. Modelling or other theoretical works were beyond the scope of this study as they are typically developed after the success of experimental measurement.

#### 1.7 Research activities

The implementation of this study has been summarised as a flowchart, as shown in Figure 1.1. This study focuses on the SET fabrication in suspended CNTs for mechanical resonator application. This work involved several steps: first, the fabrication of the suspended CNTs using the mechanical transfer technique for the first time; then, the fabrication of the double barrier on the nanotubes by Ga ion beam irradiation; and, finally, a demonstration of the SET.

The first step of this work was to deposit Palladium (Pd) as the source and drain electrode on the substrate before fabricating the suspended nanotubes.

Pd, a metal used to deposit the electrode for a device, is normally used in all device fabrication processes. However, we chose Pd compared to other metals because the contact resistance between Pd and a CNT is quite low, around 10 k $\Omega$ , which is crucial for SET fabrication. Hence, we can modulate the resistance of the nanotube to create a tunnel barrier or damage on the nanotube by FIB. Using the standard lift-off process, the electrode was fabricated at the desired thickness and gap using the standard lithography technique. Then, the nanotube was transferred to the top of the electrode using the mechanical transfer technique. The sample was characterised using a scanning electron microscope (SEM) and probed to check its current flow. If the sample had a good current flow, the resistance should be around 10 k $\Omega$  - 20 k $\Omega$ , and the next step would be to form a tunnel barrier using the Ga FIB technique. If the current flow was not good - being higher or lower than that range (10 k $\Omega$  - 20 k $\Omega$ ) - the device fabrication had to be restarted from the beginning. Finally, the sample was characterised in liquid He at a temperature of 1.5 K to check its barrier height and SET functionalities.



Figure 1.1 Research activities

#### **1.8** Overview of thesis organization

This thesis is organised into six chapters. Chapter 1 gives an overview of the research background and the motivation for the study. The originality, objectives, scope and research activities of the current work are also presented.

Chapter 2 gives an overview of the basic properties of CNTs and their possible electronic application. The chapter discusses, in brief, the principle of SETs and the possibility of coupling electron transport and mechanical motion. The challenge of fabricating suspended nanotubes and the formation of tunnel barriers are also discussed in this chapter.

In chapter 3, the fabrication process, characterisation technique and work to test the fabricated devices are described. In this work, suspended MWCNTs were fabricated using the mechanical transfer technique, which is demonstrated for the first time. The formation of tunnel barriers and the measurement setup are also explained in this chapter.

Chapter 4 describes the properties of the fabricated devices, using a single-barrier sample. This chapter discusses in detail the IV curve after irradiation at room temperature, the dose dependence at room temperature, the IV curve at low temperature, the estimation of barrier height by Arrhenius plot, the effect of barrier height on dose dependence and the diameter of the nanotubes.

In Chapter 5, the SET demonstration with a double-barrier sample is discussed. In this chapter, the Coulomb oscillation, the grey-scale plot of differential conductance, the instability in the suspended nanotube and the possible reasons for this instability were investigated.

Finally, Chapter 6 concludes the contribution of the present work and discusses future research directions.

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### LIST OF PUBLICATION

Publication related to this work: -

 Norizzawati M. Ghazali, Hiroshi Tomizawa, Noriyuki Hagiwara, Katsuya Suzuki, Abdul M. Hashim, Tomohiro Yamaguchi, Seiji Akita, and Koji Ishibashi, "Fabrication of tunnel barriers and single electron transistors in suspended multi-wall carbon nanotubes", AIP Advances 2019 9 (10): pp. 105015-1-6.

Other interest: -

 Fabian Könemann\*, Morten Vollmann, Tino Wagner, Norizzawati Mohd Ghazali, Tomohiro Yamaguchi, Andreas Stemmer, Koji Ishibashi, and Bernd Gotsmann, "Thermal Conductivity of a Supported Multiwalled Carbon Nanotube", *Journal of Physical Chemistry C*, 2019. 19 (123): pp. 12460–12465.

Conference attended: -

- 1. Norizzawati Mohd Ghazali, Hiroshi Tomizawa, Noriyuki Hagiwara, Katsuya Suzuki, Abdul Manaf Hashim, Tomohiro Yamaguchi, Seiji Akita and Koji Ishibashi "Fabrication of tunnel barrier in suspended multi-wall carbon nanotube controlled by Ga focused ion beam irradiation", The 78<sup>th</sup> JSAP Autumn Meeting, 4-7 September 2017, Fukuoka, Japan.
- 2. Norizzawati Mohd Ghazali, Hiroshi Tomizawa, Noriyuki Hagiwara, Katsuya Suzuki, Abdul Manaf Hashim, Tomohiro Yamaguchi, Seiji Akita and Koji Ishibashi "Fabrication and characterization of tunnel barriers in suspended multiwall carbon nanotubes", 43rd International Conference on Micro and Nanoengineering,18-22 September (MNE 2017), Braga, Portugal.
- 3. Norizzawati Mohd Ghazali, Hiroshi Tomizawa, Noriyuki Hagiwara, Katsuya Suzuki, Abdul Manaf Hashim, Tomohiro Yamaguchi, Seiji Akita and Koji Ishibashi "Fabrication of tunnel barriers in multi-wall carbon nanotube by Ga focused ion beam irradiation", International symposium on hybrid quantum systems (HQS 2017), 10-13 September 2017, Miyagi-Zao, Japan.
- 4. Norizzawati Mohd Ghazali, Hiroshi Tomizawa, Noriyuki Hagiwara, Katsuya Suzuki, Abdul Manaf Hashim, Tomohiro Yamaguchi, Seiji Akita and Koji Ishibashi. "Fabrication of Tunnel Barriers in Suspended Multiwall

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- Norizzawati Mohd Ghazali, Tomizawa Hiroshi, Abdul Manaf Hashim, Tomohiro Yamaguchi, Seiji Akita, and Koji Ishibashi. "Characteristics of Tunnel Barrier of Quantum Dot in Suspended Carbon Nanotube Fabricated by Focused Ion Beam", Malaysia-Japan Joint International Conference (MJJIC), 6-7 September 2016, Kuala Lumpur, Malaysia.
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- Fabian Konemann\*, Morten Vollmann, Fabian Menges, I-Ju Chen, Norizzawati Mohd Ghazali, Tomohiro Yamaguchi, Claes Thelander, Koji Ishibashi, and Bernd Gotsmann "Real-Space Scanning Probe Thermometry of Joule Self-Heating and Thermoelectric efects in Polytype III-V Nanowires and Carbon Nanotubes", Nanoscale and Microscale Heat Transfer VI – 2018, 2 - 7 December 2018, Levi, Lapland, Finland.