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2
3 **Size distributions and exposure concentrations of nanoparticles**
4 **associated with the emissions of oil mists from fastener**
5 **manufacturing processes**
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20 **Abstract**

21 The aims of the present study were set out to measure size distributions and exposure
22 concentrations of oil mist nanoparticles in three selected workplaces of the forming,
23 threading, and heat treating areas in a fastener manufacturing plant by using a
24 modified electrical aerosol detector (MEAD). The results were further compared with
25 those simultaneously obtained from a nanoparticle surface area monitor (NSAM) and
26 a scanning mobility particle sizer (SMPS) for the validation purpose. Results show
27 that oil mist nanoparticles in the three selected process areas were formed mainly by
28 the evaporation and condensation process. The measured size distributions of
29 nanoparticles were consistently in the form of uni-modal. The fraction of
30 nanoparticles deposited on the alveolar (AV) region was consistently much higher
31 than that on the head airway (HD) and tracheobronchial (TB) regions in both number
32 and surface area concentrations. However, a significant difference was found in the
33 fraction of nanoparticles deposited on each individual region while different exposure
34 metrics were used. Comparable results were found between results obtained from both
35 NSAM and MEAD. After normalization, no significant difference can be found
36 between the results obtained from SMPS and MEAD. It is concluded that the obtained
37 MEAD results are suitable for assessing oil mist nanoparticle exposures.

38 **Keywords:** nanoparticle, exposure assessment, lung deposition, modified electrical
39 aerosol detector, oil mist

40 **1. Introduction**

41 The manufacture of fasteners involves seven industrial processes, including the
42 wiredrawing, forming, threading, cleaning, heat treatment, surface treatment, and
43 packaging and shipping. Among them, the mineral oil-based metalworking fluids
44 (MWFs) are used in the three processes of the forming, threading, and heat treatment,
45 and thus might result in the emissions of oil mists to the workplace atmosphere and
46 cause workers' exposures [1,2]. Epidemiological and animal studies have indicated
47 that oil mist exposures might result in the laryngeal cancer, asthma, bronchial
48 hyper-responsiveness, lipoid pneumonia, and lung cancer [3-6].

49 In principle, machining operations would mainly generate aerosols with particle
50 sizes greater than 1 μm . However, the emissions of sub-micron and nano-sized
51 particles could still be possible [7–10]. William et al. have reported that aerosols
52 generated from an engine machining and assembly facility fell to the range from
53 0.023 μm to 0.1 μm [11]. In particular, for those involve 'hot' processes, such as
54 welding, heat treatment, and high-speed machining processes, are known to generate
55 nanoparticles [1,12–14]. It is known that MWFs are semi-volatile in nature,
56 nanoparticles could be formed through the evaporation and condensation mechanisms
57 after MWFs being "heated" during machining operations [15–16]. However, it should
58 be noted that very few studies have been conducted to address workers' exposures to
59 nanoparticles arising from oil mist emissions in workplaces which involved with the
60 use of MWFs.

61 Nanoparticles are known for particles with diameters less than 0.1 μm (or 100 nm)
62 [17]. Nanoparticles might cause serious inflammation in the deep lung because of
63 their large particle numbers and surface areas [18–20]. Recent toxicological studies
64 have suggested that they can easily penetrate cells or tissue and result in many

65 irreversible pulmonary health effects [21–23]. It has also been found that
66 nanoparticles can penetrate to the brain via nasal mucosa and olfactory buds [23]. It is
67 known that both the total surface area and total number concentrations are better
68 exposure metrics for assessing ill-health effects caused by nanoparticle exposures than
69 the metric of the total mass concentration [24–28]. In addition, ill-health effects
70 associated with nanoparticle exposures are also affected by their deposition regions in
71 the respiratory tract. Therefore, simultaneously measuring both the total surface area
72 and total number concentrations of nanoparticles exposed to different regions of the
73 respiratory tract is considered a better approach for characterizing nanoparticle
74 exposures.

75 Many instruments, such as the condensation particle counter (CPC; Model 3020,
76 TSI Inc., Shoreview, MN, USA), scanning mobility particle sizer (SMPS; Model 3934,
77 TSI Inc., Shoreview, MN, USA), electrical low-pressure impactor (ELPI; Dekati Ltd.,
78 Tampere, Finland), and nano-micro-orifice uniform deposit impactor (Nano-MOUDI;
79 Model 110, MSP Corp., Shoreview, MN, USA), have been used for assessing
80 nanoparticle exposures for workers in various industries. However, the
81 aforementioned devices can neither be used to directly measure their surface area
82 concentrations, nor to estimate exposure concentrations in different regions of the
83 respiratory tract (including the head airway (HD), tracheobronchial (TB) and alveolar
84 (AV) regions). Recently, a nanoparticle surface area monitor (NSAM; Model 3550,
85 TSI Inc., Shoreview, MN, USA) has been developed, based on the particle charging
86 characteristics of an electrical aerosol detector (EAD; Model 3070a, TSI Inc.,
87 Shoreview, MN, USA) to directly measure surface area concentrations of
88 nanoparticles deposited on both TB and AV regions of the respiratory tract [29– 30].
89 However, it should be noted that the above instrument can neither simultaneously

90 measure the surface area concentration of the HD region, nor the number
91 concentrations of the HD, TB, and AV regions. More recently, a modified EAD
92 (MEAD) has been developed by our research group to overcome the above mentioned
93 shortcomings [31–32], and the device had been successfully used in the carbon black
94 manufacturing workplaces [33].

95 The purposes of the present study were set out to use the MEAD to characterize
96 size distributions of oil mist nanoparticles and their exposure concentrations to
97 different regions of the respiratory tract, in both total surface and number
98 concentrations, for workers in fastener manufacturing industry workplaces. The above
99 results were further compared with those simultaneously obtained from SMPS and
100 NSAM for the validation purpose.

101 **2. Material and methods**

102 **2.1. Sampling sites**

103 Field sampling were conducted at the three manufacturing processes of the forming,
104 threading, and heat treatment associated with the use of MWFs. For the former two
105 processes, the involvement of both the impaction and compression would lead to the
106 increase in wire temperatures. Therefore, MWFs are used for the purpose of reducing
107 wire temperature and extending machine life. After the threading process, the treaded
108 products are quenched by passing through MWFs. Then, the products are annealed to
109 room temperature. Finally, they are tempered by raising temperatures from 650°C to
110 1,500°C to obtain products with requested hardness and toughness [34].

111 In the present study, an outdoor sampling site, located at the outside of office
112 building of the selected fastener manufacturing plant, was also selected to determine
113 the background concentration of measured nanoparticles.

114 **2.2. Sampling instruments**

115 A MEAD was used to conduct samplings for nanopartilces in the present study. The
116 MEAD was installed with a high voltage power supply (Stanford Research Systems
117 Inc., Model PS325/2500V–25W, Sunnyvale, CA, USA) to have its voltages of the ion
118 trap become variable (range: 20V–2500V). During samplings, the readings of the
119 electrometer were recorded respectively while the voltages of the ion trap were
120 consecutively set at 20V, 100V, 200V, 500V, 1000V, 1500V, 2000V, and 2500V (each
121 for ten seconds) for each run [31]. Two reference instruments were simultaneously
122 used to measure nanoparticles in order to validate results obtained from the MEAD.
123 The first one was the NSAM (TSI Inc., Model 3550, St. Paul, MN, USA) which was
124 used to measure surface area concentrations of nanoparticles deposited on both TB
125 and AV regions of the respiratory tract [30]. The second one was the SMPS (TSI Inc.,
126 Model 3936, St. Paul, MN, USA) which was used to measure the number
127 concentrations of nanoparticles of different particle sizes.

128 **2.3. *Sampling methods***

129 For all selected workplaces (including the forming, threading, heat treatment
130 processes, and outdoor sampling site), samplings were conducted for continuous four
131 days. On each sampling day for each selected workplace, one MEAD, one NSAM and
132 one SMPS were placed side-by-side at the location nearest to worker's breathing zone
133 (i.e., location ~1.5 m above the ground level). Samplings were conducted from 08:00
134 AM to 10:00 AM and from 08:00 AM to 12:00 AM to determine the outdoor
135 atmospheric background concentration and workers' daily exposure concentrations,
136 respectively. Considering workers in these three selected areas worked for 24 h per
137 day (three-shift), no workplace background concentrations could be measured.

138 **2.4. *Data analyses***

139 In the present study, a data-reduction scheme was used to retrieve the size

140 distribution of sampled nanoparticles based on readings obtained the eight preset
141 voltages of the MEAD. Detailed computation processes can be seen in our previous
142 publication [31]. The resultant size distributions were used to predict depositions of
143 nanoparticles at the H, TB, and AV regions of the respiratory tract using the UK
144 National Radiological Protection Board's (NRPB's) LUDEP Software [35]. The
145 above software was established based on ICRP 66 lung deposition models [36]. In the
146 present study, we assumed the breathing pattern of workers can be described as
147 follows:

- 148 –Breathing type: nose only
- 149 –Functional lung residual capacity: 3301 mL
- 150 –Breathing rate: 20 Breath/min
- 151 –Ventilation rate: 1.5 m³/h
- 152 –Activity level: light exercise.

153 The above criteria were the same as that prescribed for NSAM [30]. Fig. 1. shows
154 three predicted deposition curves of the HD, TB, and AV regions based on the above
155 assumptions, respectively. Here, it should be noted that the above predicted deposition
156 curves are only suitable for workers with light exercise conditions under nose-only
157 breathing conditions. The above working scenarios were quite comparable to those
158 workers in the three selected processes via our field observations.

159 **3. Results and discussion**

160 **3.1. Size distributions of nanoparticles**

161 Table 1 shows size distributions of nanoparticles (measured particle size range:
162 1–1000 nm) in the atmosphere of the three selected workplaces and the outdoor
163 ambient air. It can be seen that the count median diameter (CMD) and the
164 corresponding geometric standard deviation (σ_g) for nanoparticles of the outdoor

165 ambient air were 41.1 nm and 2.2, respectively. The above results were similar to the
166 results obtained from Heitbrink et al. and Wake [11, 37]. As shown in Fig. 2., size
167 distributions of nanoparticles were consistently in the form of the uni-modal for
168 samples collected from the forming area, threading area, and heat treating area with
169 CMD and its corresponding σ_g as 26.9 nm and 2.64, 23.2 nm and 2.86, and 22.5 nm
170 and 2.98, respectively. In an engine machining and assembly facility workplace
171 atmosphere, Heitbrink et al. found that the resultant uni-modal nanoparticles could be
172 mainly contributed by the evaporation/condensation because of MWFs being heated
173 at the interface between the tools and the components during machine operations [11].
174 At this stage, it might not be possible to explain the intrinsic differences in CMD
175 among three studied industrial processes because factors associated with the evolution
176 of aerosols in the field were very complicated (such as saturated vapor pressure,
177 surface tension, and molecular weights of the involved MWFs, etc.) [38–39].
178 However, our results are quite comparable to those conducted by Heitbrink et al. (i.e.,
179 particle size range= 20–40 nm) [11].

180 **3.2. Number concentrations and surface area concentrations of nanoparticles**

181 Table 2 shows the number and surface area concentrations of nanoparticles
182 (measured particle size range: 1–1000 nm) for the outdoor atmospheric background
183 and the three selected workplaces. The mean number concentrations for the forming
184 area, threading area, and heat treating area ($=1.42\text{--}3.47\times 10^5 \text{ \#/cm}^3$) were significantly
185 higher than that of the outdoor environment ($=0.126\times 10^5 \text{ \#/cm}^3$) ($p<0.05$). The above
186 results clearly indicate that process emissions could effectively elevate the number
187 concentrations of nanoparticles in workplace atmospheres. However, we also found
188 that their workplace concentrations fell within the range obtained from an engine
189 machining and assembly plant conducted by Heitbrink et al. ($= 0.29\text{--}4.4\times 10^5 \text{ \#/cm}^3$)

190 [11].

191 The mean number concentrations obtained from the forming area ($=2.13 \times 10^5$
192 $\#/cm^3$) was significantly higher than the threading area ($=1.42 \times 10^5 \#/cm^3$)
193 (nonparametric Mann-Whitney test, $p < 0.05$). Based on our previous study [38], the
194 measured surface temperatures on the molder of the forming machine ($=75.8 \pm 19.8^\circ C$)
195 were higher than that on the surface of the threading gear ($=69.6 \pm 17.1^\circ C$). In
196 addition, we also found that the workplace area of the threading process ($=734.4 m^2$)
197 was much larger than that of forming process ($=194.7 m^2$). Therefore, it could be
198 expected that the forming area had higher number concentrations than that of the
199 threading area by considering the generation of oil mists due to the evaporation and
200 condensation processes, and the dilution effect associated the volumes of the above
201 two workplaces. Finally, we found the heat treating area had the highest number
202 concentration among the three selected industrial processes ($p < 0.005$). In the present
203 study, the temperatures measured from those MWFs tanks used in quenching and
204 tempering steps of heat treating operations ($850\text{--}1300^\circ C$ and $650\text{--}1300^\circ C$,
205 respectively) were much higher than the temperatures measured from the other two
206 processes (as described above). Indeed, both temperatures of fluid and air would
207 affect how semi-volatile substances evaporate and condensate in the workplace
208 atmosphere [40–41]. Since the workplace temperatures of the heat treating process
209 were still less than $30^\circ C$, the highest number concentration found in the heat treating
210 process would be theoretically plausible.

211 Finally, the trends found in the measured number concentrations (as described
212 above) can also be seen in their corresponding surface area concentrations. In the
213 present study, significant differences can be found between the mean surface area
214 concentration of the outdoor atmospheric background ($=0.218 \times 10^3 \mu m^2/cm^3$) and that

215 of the concentrations of the three selected areas ($=1.11-7.48 \times 10^3 \mu\text{m}^2/\text{cm}^3$) ($p < 0.05$).
216 Moreover, workplace concentrations of the threading area ($=2.03 \times 10^3 \mu\text{m}^2/\text{cm}^3$) and
217 the forming area ($=3.06 \times 10^3 \mu\text{m}^2/\text{cm}^3$) were lower than that of the heat treating area
218 ($=5.39 \times 10^3 \mu\text{m}^2/\text{cm}^3$).

219 Furthermore, we compared the estimated number concentrations of the three
220 workplaces obtained from MEAD with that obtained from SMPS. Significant
221 differences can be found between measured values (paired t-test, $p < 0.05$) obtained
222 from MEAD and that from SMPS. In particular, values obtained from the MEAD
223 were consistently higher than that from SMPS. Considering measuring principles of
224 the MEAD was different from that of SMPS, the existence of systemic differences
225 between their measured results could be theoretically plausible. A similar result can
226 also be found in a study conducted by Woo et al. in measuring atmospheric
227 nanoparticle concentrations [42]. In this study, the results obtained from the SMPS
228 were used as the reference to normalize the values obtained from the MEAD. No
229 significant difference can be found between the measured values obtained from SMPS
230 and the corresponding normalized MEAD values (paired t-test, $p > 0.05$) (Fig. 3.). The
231 relationship between the results obtained from SMPS (i.e., x) and the normalized
232 MEAD results (i.e., y) was found as $y = 0.93x$ ($n = 18$, corrected- $R^2 = 0.74$). Therefore,
233 the number concentrations obtained from MEAD were further validated.

234 **3.3. Estimated concentrations of nanoparticles deposited on different regions of** 235 **the respiratory tract**

236 In this study, the measured size distribution data was further used to estimate both
237 the number and surface area concentrations of nanoparticles deposited on different
238 regions of the respiratory tract for the three selected workplaces. Table 3 shows the
239 estimated number concentrations (and their fractions) deposited on the three regions

240 of the HD, TB, and AV of the respiratory tract. For the forming area, the estimated
241 number concentrations for the HD, TB, and AV regions were $0.252 \times 10^5 \text{ \#/cm}^3$,
242 $0.275 \times 10^5 \text{ \#/cm}^3$, and $0.861 \times 10^5 \text{ \#/cm}^3$, respectively. For the threading area were
243 $0.191 \times 10^5 \text{ \#/cm}^3$, $0.204 \times 10^5 \text{ \#/cm}^3$, and $0.536 \times 10^5 \text{ \#/cm}^3$, respectively. For the heat
244 treating area were $0.515 \times 10^5 \text{ \#/cm}^3$, $0.517 \times 10^5 \text{ \#/cm}^3$, and $1.27 \times 10^5 \text{ \#/cm}^3$,
245 respectively. The fractions of nanoparticles deposited on the three regions, while
246 presented in sequence, were (1) forming area: AV (62%) > TB (20%) > HD (18%), (2)
247 threading area: AV (57%) > TB (22%) > HD (21%), and (3) heat treating area: AV
248 (56%) > TB (22%) and HD (22%). The above results clearly indicate that the fractions
249 of nanoparticles deposited on the AV region were much higher than that of the other
250 two regions for all selected workplaces.

251 Table 4 shows the estimated surface area concentrations (and their fractions)
252 deposited on the three regions of the HD, TB, and AV of the respiratory tract for the
253 three selected workplaces. For the forming area, the estimated surface area
254 concentrations for the HD, TB, and AV regions were $2.73 \times 10^2 \text{ \mu m}^2/\text{cm}^3$,
255 $1.64 \times 10^2 \text{ \mu m}^2/\text{cm}^3$, and $6.71 \times 10^2 \text{ \mu m}^2/\text{cm}^3$, respectively. For the threading area, they
256 were $1.37 \times 10^2 \text{ \mu m}^2/\text{cm}^3$, $0.705 \times 10^2 \text{ \mu m}^2/\text{cm}^3$, and $2.89 \times 10^2 \text{ \mu m}^2/\text{cm}^3$, respectively. For
257 the heat treating area, they were $3.91 \times 10^2 \text{ \mu m}^2/\text{cm}^3$, $1.77 \times 10^2 \text{ \mu m}^2/\text{cm}^3$, and
258 $7.34 \times 10^2 \text{ \mu m}^2/\text{cm}^3$, respectively. The fractions of nanoparticles deposited on the three
259 regions, while presented in sequence, shared the same trend as (1) forming area: AV
260 (60%) > HD (25%) > TB (15%), (2) threading area: AV (58%) > HD (28%) > TB (14%),
261 and (3) heat treating area: AV (56%) > HD (30%) > TB (14%). By comparing the
262 results shown in Table 3 and Table 4, significant differences can be found in the
263 fractions of nanoparticles deposited on each individual region while different
264 exposure metrics were adopted. Our results clearly indicate the importance for

265 simultaneously measuring both the surface area and number concentrations of
266 nanoparticles deposited on different regions of the respiratory tract for nanoparticle
267 exposure assessments.

268 Fig. 4. compares the results of the surface area concentrations deposited on both
269 the TB and AV regions obtained from MEAD with that obtained from NSAM. For the
270 concentrations estimated for the TB region, the results obtained from the NSAM for
271 the forming area, threading area, and heat treating area were $1.19 \times 10^2 \mu\text{m}^2/\text{cm}^3$,
272 $0.562 \times 10^2 \mu\text{m}^2/\text{cm}^3$, and $1.27 \times 10^2 \mu\text{m}^2/\text{cm}^3$, respectively. The above results were
273 quite comparable to those obtained from MEAD ($= 1.64 \times 10^2 \mu\text{m}^2/\text{cm}^3$, 0.705×10^2
274 $\mu\text{m}^2/\text{cm}^3$, and $1.77 \times 10^2 \mu\text{m}^2/\text{cm}^3$, respectively) (t-test, $p > 0.05$). The same trend can
275 also be found for the concentrations estimated for the AV region (NSAM= 6.73×10^2
276 $\mu\text{m}^2/\text{cm}^3$, $2.65 \times 10^2 \mu\text{m}^2/\text{cm}^3$, and $5.30 \times 10^2 \mu\text{m}^2/\text{cm}^3$, respectively; and
277 MEAD= $6.71 \times 10^2 \mu\text{m}^2/\text{cm}^3$, $2.89 \times 10^2 \mu\text{m}^2/\text{cm}^3$, and $7.34 \times 10^2 \mu\text{m}^2/\text{cm}^3$, respectively)
278 (t-test, $p > 0.05$). Considering both NSAM and MEAD sharing the same measuring
279 principles (i.e., particle charging efficiency and particle electrical mobility),
280 comparable results obtained from both instruments could be theoretically expectable.

281 4. Conclusions

282 We found that size distributions of nanoparticles were consistently in the form of
283 uni-modal for the three selected process areas. It could be mainly contributed by the
284 evaporation and condensation processes of MWFs. For both number and surface area
285 concentrations, the fractions of nanoparticles deposited on the AV region were much
286 higher than that of the other two regions of the TB and HD for all selected workplaces.
287 However, a significant difference was found in the fractions of nanoparticles
288 deposited on each individual region of the respiratory tract while different exposure
289 metrics were adopted. Our results clearly indicate the importance for simultaneously

290 measuring both the surface area and number concentrations of nanoparticles deposited
291 on different regions of the respiratory tract for nanoparticle exposure assessments. In
292 the present study, results obtained from both NSAM and MEAD were quite
293 comparable. In addition, no significant difference can be found between the measured
294 values obtained from SMPS and the corresponding MEAD values after being
295 normalized. The above results clearly indicate that the measured MEAD results would
296 be theoretically plausible.

297

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301 **REFERENCES**

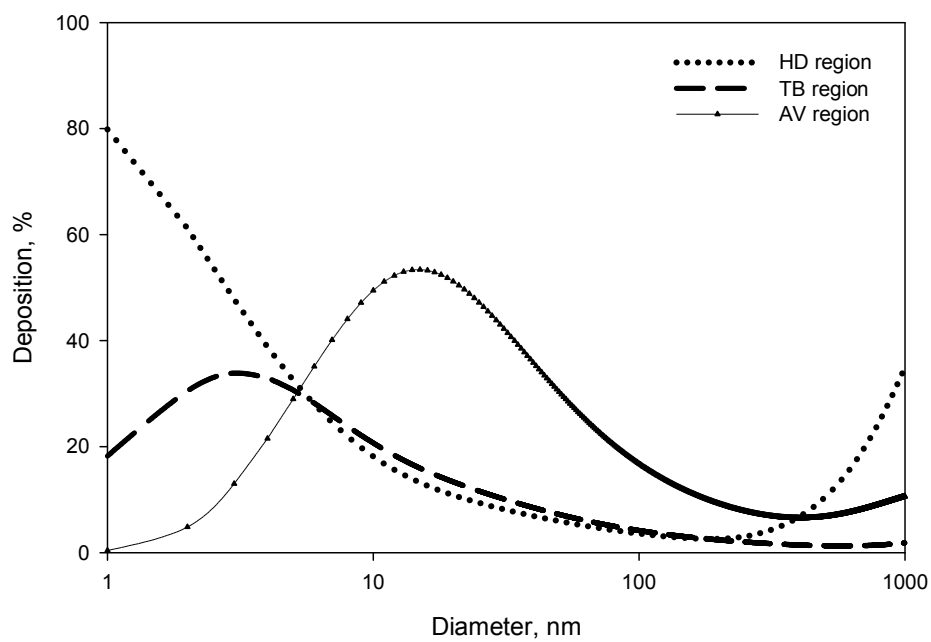
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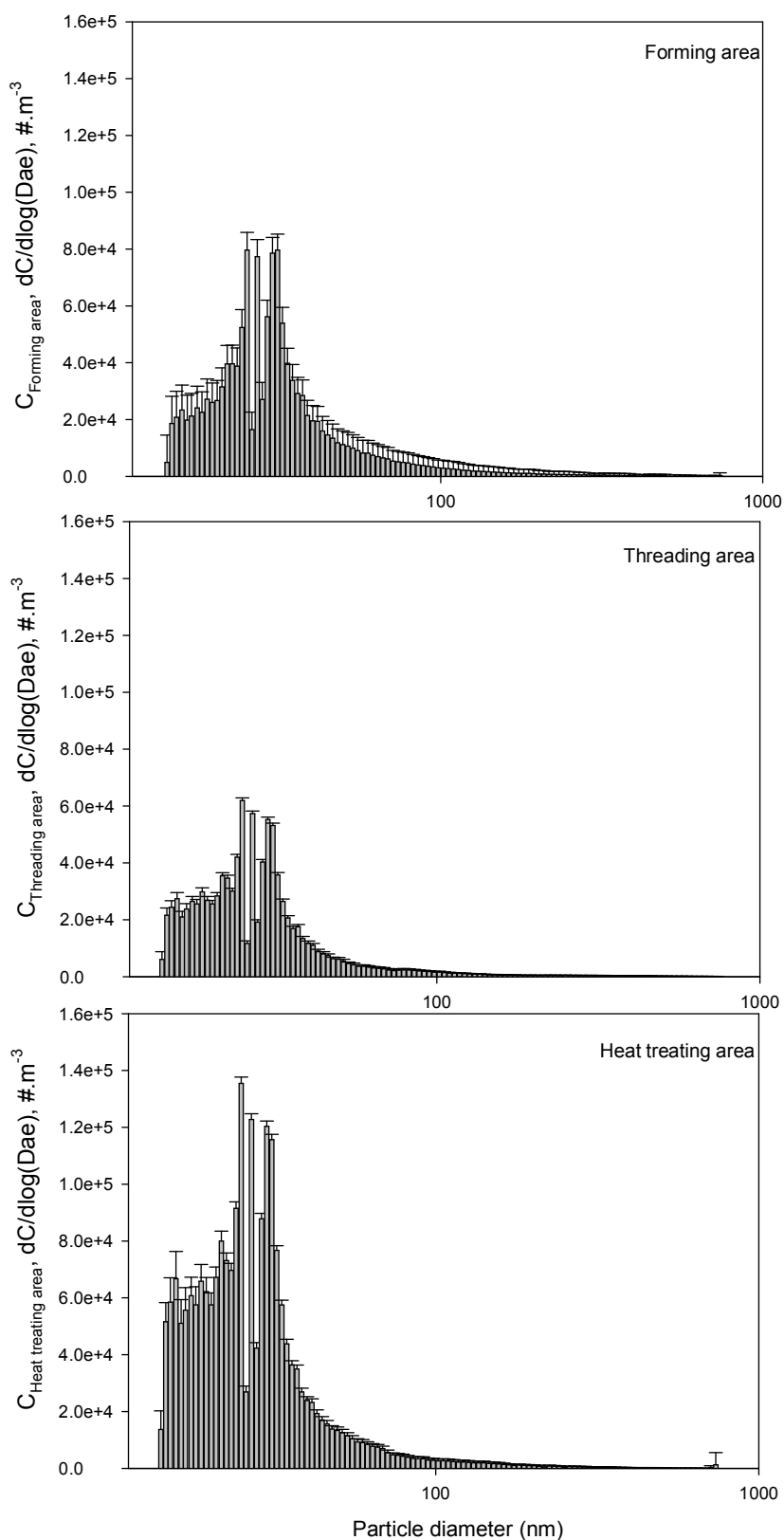
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425 Fig. 1. Calculated particle deposition curves as a function of particle size for the Head

426 Airway (HD), Tracheobronchial (TB), and Alveolar (AV) regions of a human lung

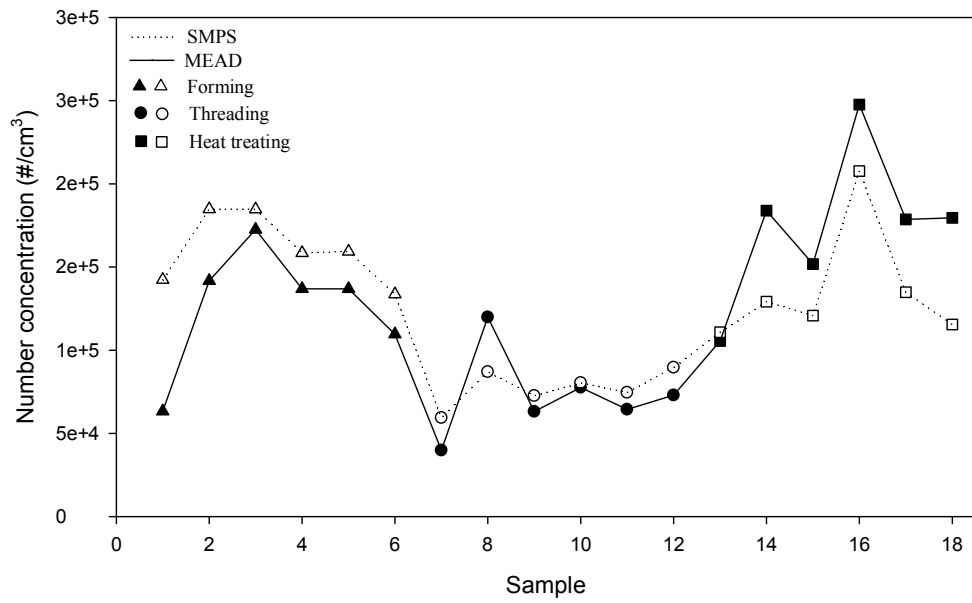
427 (based on the model given in ICRP [36]).



428

429 Fig. 2. Particle number-based size distributions in the three selected area were

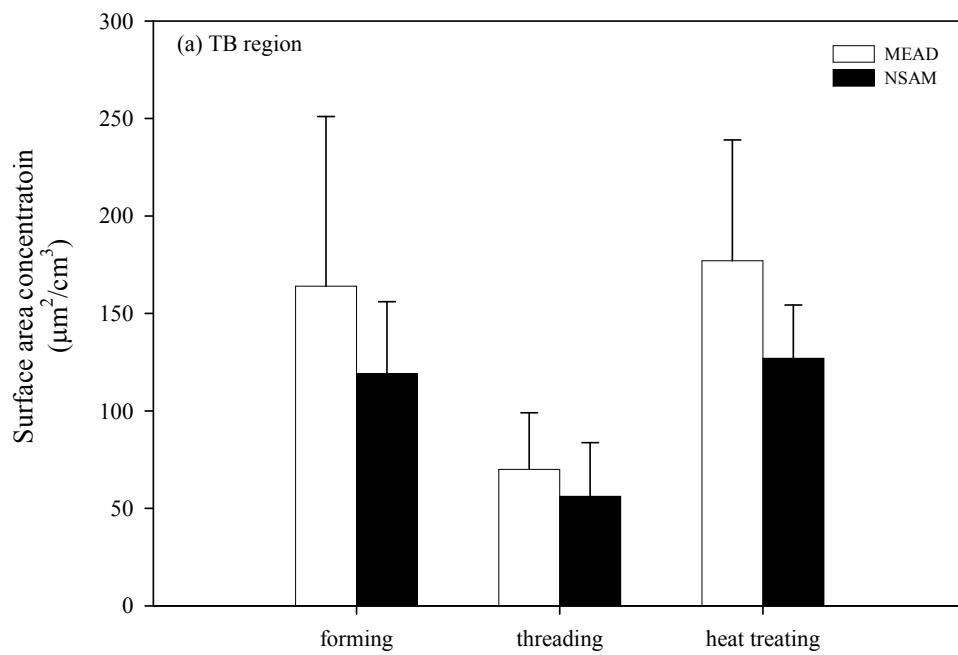
430 measured by MEAD in activity.



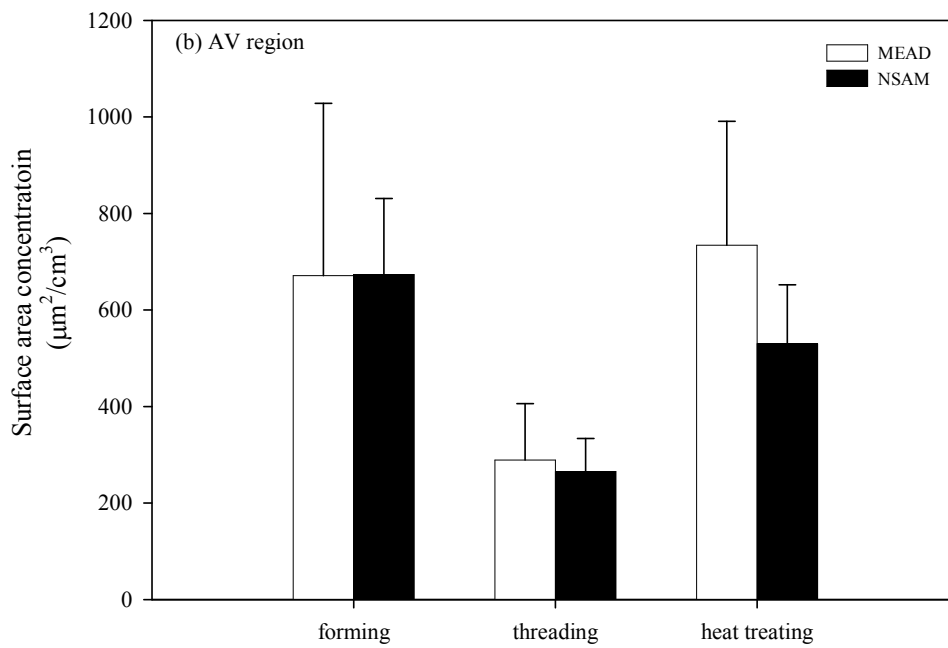
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432 Fig. 3. Comparing number concentrations obtained from SMPS with that from

433 MEAD after being normalized.



434



435

436 Fig. 4. Confirmations of nanoparticles deposited in (a) TB region and (b) AV region

437 were measured by the MEAD and NSAM.

438 Table 1 Number-based size distributions of nanoparticles (1–1000 nm) in the selected
 439 workplace were measured by MEAD (n=6).

work area	Number-based size distribution (nm)	
	CMD(range)	σ_g
forming	26.9(25.3–30.1)	2.64
threading	23.2(21.1–25.2)	2.86
heat treating	22.5(20.3–25.2)	2.98
Ambient*	41.1	2.21

440 *: n=1

441

442

443 Table 2 Estimated total number concentrations (10^5 \#/cm^3) and total surface area
 444 concentrations ($10^3 \mu\text{m}^2/\text{cm}^3$) for nanoparticles (1–1000 nm) found in the selected
 445 work area (n=6)

work area	Total number concentration (10^5 \#/cm^3)		Total surface area concentration ($10^3 \mu\text{m}^2/\text{cm}^3$)	
	mean±SD	range	mean±SD	range
	forming	2.13±1.05	1.23–3.35	3.06±1.14
threading	1.42±0.572	0.772–2.33	2.03±0.733	1.11–3.33
heat treating	3.47±1.22	2.05–4.80	5.39±1.46	3.18–7.48
Ambient*	0.126	–	0.218	–

446 *: n=1

447 Table 3 Estimated number concentrations ($10^5 \#/\text{cm}^3$) deposited in the HD, TB, and
 448 AV regions of the respiratory tract for nanoparticles (1–1000 nm) found in the
 449 selected workplace (n=6)

work area	Total deposited conc.	HD		TB		AV	
		Conc.	Fraction (%)	Conc.	Fraction (%)	Conc.	Fraction (%)
forming	1.38±1.07	0.252±0.203	18	0.275±0.221	20	0.861±0.643	62
threading	0.922±0.372	0.191±0.083	21	0.204±0.082	22	0.536±0.212	57
heat treating	2.27±0.791	0.515±0.176	22	0.517±0.183	22	1.27±0.454	56

450

451

452 Table 4 Estimated surface area concentrations ($10^2 \mu\text{m}^2/\text{cm}^3$) deposited in the HD, TB,
 453 and AV regions of the respiratory tract for nanoparticles (1–1000 nm) found in the
 454 selected workplace (n=6)

work area	Total deposited conc.	HD		TB		AV	
		Conc.	Fraction (%)	Conc.	Fraction (%)	Conc.	Fraction (%)
forming	11.1±5.90	2.73±1.45	25	1.64±0.873	15	6.71±3.57	60
threading	4.97±2.01	1.37±0.551	28	0.705±0.298	14	2.89±1.17	58
heat treating	13.2±4.57	3.91±1.37	30	1.77±0.627	14	7.34±2.57	56

455