More on the Dynamics of Dust Generation: The Effects of Mixing and Sanding Chrysotile, Calcium Carbonate, and Other Components on the Characteristics of Joint-Compound Duffs

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An ongoing research effort designed to reconstruct the character of historical exposures associated with use of chrysotile-containing joint compounds naturally raised questions concerning how the character (e.g. particle size distributions) of dusts generated from use of recreated materials compares to dusts from similar materials manufactured historically. This also provided an opportunity to further explore the relative degree that the characteristics of dusts generated from a bulk material are mediated by the properties of the bulk material versus the mechanical processes applied to the bulk material by which the dust is generated. In the current study, the characteristics of dusts generated from a recreated ready mix and recreated dry mix were compared to each other, to dusts from a historical dry mix, and to dusts from the commercial chrysotile fiber (JM 7RF3) used in the recreated materials. The effect of sanding on the character of dusts generated from these materials was also explored. Dusts from the dry materials studied were generated and captured for analysis in a dust generator-elutriator. The recreated and historical joint compounds were also prepared, applied to drywall, and sanded inside sealed bags so that the particles produced from sanding could be introduced into the elutriator and captured for analysis. Comparisons of fiber size distributions in dusts from these materials suggest that dust from commercial fiber is different from dusts generated from the joint compounds, which are mixtures, and the differences persist whether the materials are sanded or not. Differences were also observed between sanded recreated ready mix and either the recreated dry mix or a historical dry mix, again whether sanded or not. In all cases, however, such differences disappeared when variances obtained from surrogate data were used to better represent the ‘irreducible variation’ of these materials. Even using the smaller study-specific variances, no differences were observed between the recreated dry mix and the historical dry mix, indicating that chrysotile-containing joint compounds can be recreated using historical formulations such that the characteristics of the modern material reasonably mimic those of a corresponding historical material. Similarly, no significant differences were observed between dusts from sanded and unsanded versions of similar materials, suggesting (as in previous studies) that the characteristics of asbestos-containing dusts are mediated primarily by the properties of the bulk material from which they are derived.

Keywords: asbestos; chrysotile; dust characteristics; dust generation; fiber size distributions; joint compound; mixing; sanding

INTRODUCTION

It has previously been shown that the cancer potency of an asbestos fiber is a function of the fiber’s size...
and mineral type (reviewed in Nicholson, 1986; Berman and Crump, 2003, 2008a,b). However, an asbestos exposure metric that fully explains the observed cancer incidence across available epidemiology studies remains to be identified. Thus, better characterization of relevant historical exposures is needed before candidate metrics can be adequately evaluated (e.g. Berman and Crump, 2008a,b; Berman, 2011).

In this context, an exposure metric is the weighted set of size categories (lengths, widths, and aspect ratios) that are defined in the counting rules of an analytical method. With rare exceptions, published epidemiology studies report exposures only as the total concentration of fibers satisfying a single metric: the phase contrast microscopy (PCM) metric. As described in NIOSH Method 7400 (NIOSH, 1994a), the PCM metric is the set of all structures >5 μm with an aspect (length-to-width) ratio of ≥3 when viewed using PCM. Yet, to adequately explore the effects of fiber size and type, both the distributions of and a broader range of fiber sizes, shapes, and types need be included in each characterization of exposure.

Importantly, as used above and throughout (when referring to the microscopic particles found in asbestos-containing dusts), the term ‘fiber’ is intended to include both fibers and bundles (structures composed of bound parallel fibers), which may either represent isolated (primary) structures or components of more complex clusters (overlapping fibers or bundles arranged in random orientation) and matrices (fibers or bundles partially embedded in a non-asbestos particle) that are typically observed in asbestos dusts (ISO, 1995). In contrast, the term ‘structure’ will be used to collectively represent fibers, bundles, clusters, and/or matrices.

Because opportunities for reconstructing historical asbestos exposures by the traditional approach (i.e. reanalyzing archived air filters from environments of interest) is limited by the availability of samples, alternate approaches have also been proposed. These include regenerating historical dusts in the laboratory by applying the Modified Elutriator Method—MEM (Berman and Kolk, 1997, 2000) to appropriately selected bulk materials (Berman, 2010a,b).

The viability of using the MEM to reconstruct dusts exhibiting the character of historical fiber exposures relies on the assumption that the characteristics of an asbestos dust are determined primarily by the properties of the bulk material from which the dust derives rather than the mechanical processes by which the dust is generated. There is a good theoretical basis justifying this assumption; as long as mechanical processes do not supply sufficient energy to melt or break the individual crystals (fibers) within a composite, the size distribution of the individual crystals will be preserved when the composite is disaggregated (Wylie and Schweitzer, 1982; Turcotte, 1986; Wylie, 1993; reviewed in Berman, 2010a). Fiber studies conducted to date also reinforce the assumption’s validity (e.g. Berman, 2000, 2010a,b). However, some studies of asbestos-free materials suggest that mechanical forces affect the size distribution of (non-fibrous) particles in generated dusts (e.g. Koponen et al., 2009, 2011; Göhler et al., 2010; Wohlleben et al., 2011). Therefore, additional studies characterizing the effects of mechanical processing on fiber size would further inform this issue. Importantly, the kinetics (rate) of dust generation is neither the focus of these studies nor the current study: changes in mechanical force will always affect the kinetics of dust generation.

Similar questions also arose during an ongoing research effort designed to reconstruct the character of historical fiber exposures associated with use of chrysotile-containing joint compound (Brorby et al., 2008, 2010; Bogen et al., 2011; Jones et al., 2011; Sheehan et al., 2011; Simmons et al., 2011). As part of that effort, two types of chrysotile-containing joint compounds were recreated based on the original formulations that were used by Georgia-Pacific LLC’s (GP’s) predecessors to manufacture these materials in the late 1960s (Brorby et al., 2008). One is a joint system cement (dry mix), which is a dry product that must be mixed with water prior to use, and one is a ready mix, which is a moist putty that is ready for use straight out of its container. These materials were recreated to conduct experiments designed to better characterize dusts generated during their use and any attendant chrysotile-related exposures.

One question that naturally arises from study of recreated joint compounds is how the characteristics of dusts generated by handling and use of such materials compare to dusts from similar materials manufactured historically. Of particular interest, for example, is the nature of the size, shape, and complexity of the chrysotile fibers that occur in the dusts generated from these materials. It is also of interest to consider: (i) whether the physical mixing of a commercial fiber product (purchased from an asbestos mill) with the other components of joint compound might change the distribution of fiber sizes in dusts generated from these materials and (ii) what changes in fiber size might also be associated with the application and sanding of these recreated joint compounds. These latter two questions, which involve the effects
of physical processing, parallel those identified at the beginning of this Introduction.

In the current study, the dusts generated from the recreated joint compounds were characterized and compared to each other and to dusts generated both from samples of what has been represented to be historical joint compound (a dry mix referred to herein as ‘Bestwall’) and from the commercial fiber (JM 7RF3) used as the source of chrysotile when recreating these joint compounds. The effect of sanding on the characteristics of dusts generated from these materials was also explored.

**BACKGROUND**

Conducting valid comparisons across characteristics of bulk materials (or the characteristics of dusts generated from bulk materials) is difficult for a number of reasons (e.g. Berman and Kolk, 1997, 2000; Berman, 2000, 2010a,b; Gerlach and Nocerino, 2003). First, because they are composed of particles of finite size, at a sufficiently small scale, all bulk materials are necessarily heterogeneous (Gerlach and Nocerino, 2003). Moreover, the types of materials of interest in this study are heterogeneous mixtures of particles and fibers of differing sizes, shapes, and composition. Such characteristics place limitations on the ability to construct representative samples and subsamples, which leads to irreducible variation that has also been called ‘fundamental error’ (Gerlach and Nocerino, 2003). In fact, if not handled, sampled, or sub-sampled properly, even larger sources of variation and bias can be introduced into samples of such materials. Therefore, to promote valid comparison, not only must such materials be handled, sampled, and sub-sampled properly, but the irreducible variation caused by limitations in the homogeneity of sets of samples representing each individual material must be adequately addressed during data analysis and interpretation.

It has also been suggested (Berman, 2010a,b) that the characteristics of bulk materials containing asbestos (and their associated dusts) vary irreducibly to an even greater degree than expected from fundamental error alone. This is due to spatial variation in ore deposits from which the asbestos derives, which translates into temporal variation in the commercial fiber products sold by mines and corresponding variation in the asbestos characteristics of other commercial products in which the commercial fiber has been incorporated. Although classification methods used in a mill to separate fiber from ore results in grades and subgrades with distinguishable characteristics between product lines, based on the results of the Berman studies, such separation remains sufficiently coarse to result in commercial products that nevertheless exhibit substantial variation in fiber size distributions over time.

The above affects both the nature of questions to be asked when comparing samples of dusts generated from differing materials and the appropriate procedures to be used for conducting such comparisons. For example, the appropriate question to ask when comparing dusts from two different bulk materials is whether variation between the samples representing each material exceeds the irreducible variation observed among samples representing either material alone; simply finding an absolute difference between individual samples or between sets of samples from different materials does not necessarily mean that the materials are in fact different. In an earlier study (Berman, 2010a,b), for example, when comparing across samples representing factory dusts at the textile plant, laboratory dusts generated from textile grade fiber, and laboratory dusts generated from raw ore, nearly every sample was found to vary significantly from each of the others, whether representing the same or different materials. Clearly, therefore, only when differences between samples representing different materials are larger than the variation observed within materials, can it be concluded that the materials are different. The consequence in that study was that chi-square tests could not be used to compare materials; Mann–Whitney rank sum tests (Devore, 2004) were used instead (discussed in Berman, 2010a,b).

The issues identified above, which affect how valid comparisons of interest need to be conducted, are addressed further in Materials and Methods.

**MATERIALS AND METHODS**

**Materials**

Both the ready mix and the dry mix evaluated in this study were recreated in the manner previously described (Brorby et al., 2008). Samples analyzed to represent these materials were each split from the homogenized whole of the material originally prepared. The samples of 7RF3 commercial fiber evaluated in this study were split from a homogenized split of the sample of commercial fiber originally used to recreate the dry and ready mixes. 7RF3 fiber was originally obtained as a commercial product from the Johns-Manville mine in Asbestos, Quebec. The Bestwall samples evaluated in this study were split following homogenization from the sample provided by GP.
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**Methods**

Procedures and equipment used for homogenizing and splitting materials and for dust generation in this study are described briefly below and in greater detail in the MEM (Berman and Kolk, 1997, 2000). The procedures are consistent with recommendations provided in the Gerlach and Nocerino (2003) guidance for handling and sampling bulk materials. Despite being based on a feasibility study completed >10 years before the Gerlach and Nocerino (2003) guidance, the MEM was designed to address the issues identified in the 2003 guidance concerning difficulties with handling and sampling bulk materials. For this reason, the method in fact incorporates use of the same procedures and equipment (to the extent available at the time) that are now recommended in the guidance. Therefore, its use should be considered appropriate for this study.

**Homogenization and splitting**

Because unconsolidated bulk materials become vertically segregated by size over time with even minimal disturbance (Berman and Kolk, 1997; Gerlach and Nocerino, 2003), it is nearly impossible to collect a representative aliquot simply by sub-sampling. Instead, bulk samples were homogenized by subjecting each entire sample to multiple passes through a riffle splitter while recombining the splits between each pass. To derive a representative aliquot of the desired size (usually between 40 and 80 g), each material was then sequentially divided into halves by sequentially passing an uncombined split of the material (from a previous pass) through the same riffle splitter until the final splits are of the desired size. The procedure is described in greater detail in Chapter 8 of Berman and Kolk (1997) and a photograph of a riffle splitter is presented in Figure 8-4 of the same document.

**The dust generator-elutriator**

A schematic representation of the dust generator-elutriator designed for the MEM (Berman and Kolk, 2000) is provided in Figure A-1 of that document and reproduced as Fig. 1 here. Appropriately sized samples are introduced into a tumbler, which is a 1-inch (2.5 cm) square in cross-section and 12 inches (30 cm) long. The tumbler is situated within (and draws air from) a humidity-controlled chamber. To generate a representative dust sample, air is drawn through the tumbler while it is rotated at between 15 and 60 r.p.m. The dust-laden air is then passed through a connector tube to the bottom of a vertical elutriator: a cylinder that is 4 inches (10 cm) in diameter and 4 ft (120 cm) long. Air flow (0.3–0.4 cm s⁻¹) within the elutriator is adjusted so that only respirable-sized particles will be transported up the tube to be captured on sample filters over each of two exit openings at the top; larger particles settle to the bottom. Two 25-mm filter cassettes are mounted on each of two sliding mechanisms allowing filter cassettes over each exit opening to be sequentially replaced with minimal disturbance of the airflow regime through the elutriator. One opening (the ‘IST’ opening) is situated over an isokinetic sampling tube that extends into the elutriator. Samples collected over this tube provide representative samples of respirable-sized particles from the original sample, which are uniformly loaded on the filter. Samples collected over the second (‘ME’) opening also pick up respirable-sized particles but not under isokinetic conditions;
approximately 90% of the airflow from the elutriator moves through this latter opening.

**Dust generation**

So that the effects of sanding could be evaluated, dusts were generated both directly from samples of Bestwall (a dry mix) and recreated dry mix and from the particles generated from these materials (as well as the recreated ready mix) following mixing, application, drying, and sanding. To create the particles from sanding, splits of homogenized samples of Bestwall and the dry mix were prepared for use by mixing with water and (along with the ready mix) applying each to individual (30 cm by 30 cm) sections of drywall. After drying, the drywall was placed inside two re-sealable (Ziploc®) bags along with a sander and the applied joint compound was sanded from the drywall (by holding the sander through the walls of the sealed bags). After sanding, the sealed bags of joint compound dust were shipped to EMS Laboratories (Pasadena, CA, USA), so that the dust produced during sanding could be removed from the bag, homogenized, and split using the procedures of the MEM (Berman and Kolk, 1997, 2000) to create subsamples suitable for dust generation. Splits of the original Bestwall and recreated dry mix powders (along with splits of the 7RF3 fiber) were also sent to EMS Laboratories to create subsamples suitable for dust generation. Ultimately (with one exception), three samples of each of six materials were prepared for dust generation:

- Sanded Bestwall (SBW)
- Unsanded Bestwall (UBW)
- Sanded dry mix (SDM)
- Unsanded dry mix (UDM)
- Sanded ready mix (SRM)
- (Unsanded) 7RF3 fiber (7RF3).

Regarding the one exception, the small amount of UBW available for this study limited what could be applied and sanded. As a result, only two SBW samples were produced for analysis.

Dusts were generated and prepared for analysis in this study using the equipment and procedures of the MEM (Berman and Kolk, 1997, 2000). The method has been shown to reasonably reproduce the characteristics of dusts generated when bulk materials similar to those analyzed are handled in the field (Berman, 2000, 2010a). Thus, briefly

- Appropriately sized splits (between 5 and 10 g for commercial fiber and between 30 and 60 g for sanded and unsanded joint compound) of each sample of each material (in separate runs) were placed in the tumbler of a dust generator-elutriator (described in the MEM) and tumbled in a humidity-controlled air stream;
- The air stream with entrained dust then passes through the vertical elutriator of the device to separate out the fraction of particles in the respirable range
- The respirable range dust was collected on mixed-cellulose ester (MCE) filters at the top of the elutriator and
- Dust-laden filters were weighed and prepared by direct-transfer and analyzed by transmission electron microscopy (TEM) in the manner described below.

**Sample analyses**

Filter samples were analyzed by TEM using ISO Method 10312 (ISO, 1995). The stopping rules of the method were modified to assure adequate sensitivity for the requirements of this study. The goal was to count sufficient fibers to assure at least 10 fibers (on average) would be observed across size categories of interest. Thus, five grids (rather than the usual two) were prepared from each filter (one from the center and one from half-way between the center and edge at locations spaced 90° from one another around the filter) in the manner described in ISO Method 10312, and counts were divided evenly across the five grids.

The stopping rules were also modified to require that 450 grid openings be scanned at a magnification of $10,000 \times$ or a scan would be completed on the grid opening on which the last of the following obtained:

- The 50th fiber between 5 and 10 μm in length was observed,
- The 125th fiber between 10 and 40 μm in length was observed, or
- The 15th fiber $>40$ μm was observed.

A separate scan at $\sim \times 20,000 \times$ was also conducted, for which scanning was completed on the grid opening on which the 50th fiber $>0.5$ μm was observed. In this manner, it could be reasonably assured that almost all size categories of interest would be quantified. Finally, counting rules were also modified to include all fibers with an aspect (length-to-width) ratio $>3$ (instead of five as specified in the method).

**Statistical approach**

**Determining fiber size distributions.** Because each sample represents a homogenized split of the same material, the sizes of fibers (or particular types...
of fibers) coming from the separate samples can reasonably be expected to represent the same distribution. This consideration was also examined formally (see below). Therefore, size distributions (i.e. estimates of the relative fraction of fibers represented by each size category for each fiber type of interest) were generated for each material in the following manner.

For each $i$th sample filter, define:

\[ A_{ji} = \text{the relative area of the } i \text{th filter examined for fibers in the } j \text{th size category (arbitrary units)}; \]

\[ n_{ji} = \text{the number of fibers in the } j \text{th size category on the } i \text{th filter (number)}; \]

\[ \lambda_{ji} = \text{the density of the } j \text{th size category of fibers on the } i \text{th filter (str/unit area)}; \]

\[ \lambda_i = \text{the density of all fibers on the } i \text{th filter (str/unit area)}; \]

\[ f_{ji} = \frac{\lambda_{ji}}{\lambda_i} \]

The fraction $F_j$ of all fibers in each size category $j$ among samples pooled from each material was estimated as:

\[ F_j = \frac{\sum f_{ji} \times \lambda_i}{\sum \lambda_i} \]  

where $F_j$ in equation (4) can be seen to be a weighted mean of the $f_{ji}$ across samples using weights $w_i = \lambda_i / \sum \lambda_i$.

Ultimately, fibers observed in each sample (or fibers observed among pooled samples for each material) were categorized into sets of 15 mutually exclusive bins that were each defined by a range of lengths ($L$) and widths ($W$) collectively covering the entire range of fiber sizes observed. Length categories included in this analysis were $L < 5 \mu m$, $5 \mu m \leq L < 10 \mu m$, $10 \mu m \leq L < 20 \mu m$, $20 \mu m \leq L < 40 \mu m$, and $40 \mu m \leq L$. Width categories were $W \leq 0.25 \mu m$, $0.25 \mu m < W \leq 0.4 \mu m$, and $0.4 \mu m < W$. Counts from the individual categories were also combined to estimate fractions for the individual length categories (all widths combined) and individual width categories (all lengths combined). The goal was to compare the sets of material-specific $F_j$ to one another.

Separate size distributions were generated from counts of primary fibers (a primary fiber is a fiber around which an imaginary boundary can be drawn that crosses no part of the fiber and that separates it from all other fibers on the viewing screen) and from counts of total fibers (which includes fibers that are components of more complex structures). The relative fraction of primary structures (of all sizes) that are matrices (ISO, 1995) and the relative fraction of fibers (of all sizes) that are embedded in matrices also were determined.

**Comparing fiber size distributions**

Before comparing size distributions across materials, chi-square tests were used to compare observed fiber counts $n_{ji}$ for each size category of each sample of a material to expected counts $n_{eji}$ calculated in the manner described previously (Berman, 2010a; equation 6) as the product of the area scanned for that size category, the total fiber load on the filter, and the fraction of fibers in that size category estimated for each material:

\[ n_{eji} = F_j \times \lambda_i \times A_{ji}. \]  

This was done simply to evaluate whether sample sets representing each individual material exhibited the expected extra-Poisson variability, which in turn would necessitate consideration of irreducible variation when comparing between materials.

Because extra-Poisson variability within data sets was confirmed (see Results), size distributions across materials were ultimately compared using a one-way analysis of variance (ANOVA) conducted on length- or width-category-specific sets of log-transformed values representing the fraction of total fibers satisfying the dimensional criteria for each specific size category (Selvin, 1995). Separate analyses were conducted on counts of primary fibers and total fibers (which include fibers that are components of more complex structures). ANOVA tests were performed with all six materials included, with unsanded 7RF3 fiber (7RF3) excluded, and with both 7RF3 and sanded ready mix (SRM) excluded. To account for conducting multiple independent tests (considering multiple size categories simultaneously), $P$-values adjusted using Hommel’s Bonferroni-type procedure to address the performance of multiple independent tests (Wright, 1992) are denoted as $P_{\text{adj}}$ in Results.

To better account for the full range of irreducible variability expected to be exhibited by each material, ANOVA tests were conducted both using estimates of sample variance derived in the current study and using variance estimates derived from surrogate
data. The surrogate data used for this purpose were the combined counts from 84 samples of airborne dusts collected in a South Carolina textile factory, which have been previously described (Dement et al., 2008; Berman, 2010a). These samples capture the temporal variation likely introduced into asbestos products from variation in the commercial fiber purchased from a mine (Berman, 2010a,b); as there is no reason to expect otherwise, it was assumed for this study that the variation associated with (textile) Grade 3 fiber would be roughly equivalent to what would also be associated with Grade 7 fiber (the type used in joint compound manufacturing). Sample variances were calculated from the surrogate data using log-transformed values representing the fractions of total fibers satisfying the dimensional criteria for each of the same length and width categories evaluated in the present study.

Material-specific fractions of fibers in five length categories were also plotted along with their corresponding two tail 95% confidence limits (assuming approximate normality of the log-count data) derived using either from the variance estimates from this study or from the surrogate data.

Effects of matrices

With the exception of 7RF3 (which is commercial chrysotile fiber), all the other five materials studied here are composed of chrysotile mixed within a matrix of non-asbestos particles (primarily calcium carbonate with small amounts of binders and anti-fungal agents). Therefore, it is interesting to consider both the relative abundance of microscopic matrices (non-asbestiform particles containing chrysotile fibers) observed among primary structures and the fraction of chrysotile fibers observed to be embedded within such matrices when comparing these materials.

To accomplish the above, the ratio of the number of primary matrices to total primary structures was determined for each of the i samples of each material. Similarly, the ratio of the number of fibers observed to be components of matrices to the total number of fibers was also determined. As with comparisons of fiber size distributions, differences between the relative fraction of primary structures that are matrices and the relative number of fibers that are found in matrices across materials were compared by one-way ANOVA.

RESULTS

Size distributions

The mean relative frequencies of variously sized fibers in dusts generated from each of the materials evaluated in this study are depicted in Fig. 2 for primary fibers and Fig. 3 for total fibers. To facilitate use of the data, numerical values for these frequencies are also presented in an Appendix. As can be seen in the Figure (after accounting for the adjustment to frequency performed on the three width categories of the shortest length category to allow all categories to be reasonably depicted on the same figure), ~80 to 95% of either the primary or the total fibers observed in these dusts are short (L < 5 µm), and of these, >80% are thin (w ≤ 0.25 µm). It is also apparent both that the abundance of fibers decreases regularly with increasing length and that the thickness of fibers increases somewhat with increasing length for all the materials depicted.

Comparison of size distributions across materials

As expected, results of chi-square comparisons between samples within each of the six materials showed significant extra-Poisson variability (most of the within material comparisons showed P ≤ 0.001 for primary fibers and all the comparisons for total fibers showed P values that were substantially smaller). This simply confirms that valid comparisons between materials require consideration of the irreducible variation exhibited within materials (Kramer and Schmidhammer, 1992; Walters, 2000).

Figure 4 presents the pooled (average) relative frequencies of total fibers and bundles, the Fi, for the indicated length categories (all widths combined within lengths) for each of the six materials evaluated. Because only comparisons across materials within each length category are being compared here, the relative frequencies of structures for each material were normalized to that for sanded Bestwall within each length category; this allowed data from all length categories to be better represented in a single figure. Confidence intervals in the figure were constructed using variances observed across samples in the current study. Figure 5 is identical to Figure 4 except that confidence intervals were constructed using variances derived from the surrogate data.

Comparing confidence intervals between Figs. 4 and 5, it can be seen that those based on surrogate data are almost uniformly larger than those derived from the study-specific data. This was expected because samples within the sets representing each material are homogenized splits of one another, which means that they exclude the temporal variation in the commercial fiber from which they were manufactured. Therefore, they may not adequately reflect the true irreducible variation within each material. As an
Comparison of size distributions across materials. The mean relative frequencies of variously sized categories were compared by one-way ANOVA. That is, the relative fraction of primary structures served to be components of matrices to the total material. Similarly, the ratio of the number of fibers observed among primary matrices to total primary structures was evaluated in the present study.

Effects of matrices. With the exception of 7RF3 (which is commercial for this study that the variation associated with asbestos dusts collected in a South Carolina textile factory, the combined counts from 84 samples of airborne material-specific fractions of fibers in five length categories to be reasonably depicted on the same Figure). Confidences intervals were calculated from the variance estimates derived using either from the variance estimates from the surrogate data used for this purpose were constructed using variances derived from the surrogate data. The surrogate data used for this purpose were constructed using variances observed across samples in the current study. Figure 5 is identical to Figure 4 except that confidence intervals were constructed using variances derived from the surrogate data.

More on the dynamics of dust generation. Fraction of primary fibers and bundles by size category. Frequencies in these 3 width categories (all with L<5 um) were divided by 10.

Fig. 2. Fraction of primary fibers and bundles by size category.

Fraction of total fibers and bundles by size category. Frequencies in these 3 width categories (all with L<5 um) were divided by 10.

Fig. 3. Fraction of total fibers and bundles by size category.
Fig. 4. Relative frequencies of total fibers and bundles in indicated length categories with confidence intervals defined using study data (normalized in each length category to sanded Bestwall).

Fig. 5. Relative frequencies of total fibers and bundles in indicated length categories with confidence intervals defined using surrogate data (normalized in each length category to sanded Bestwall).
extreme example, consider the near zero width of the confidence interval for the frequency of structures in Sanded Bestwall between 5 and 10 μm in length. This is a consequence of the nearly identical frequency estimates derived from the (only) two samples available for this material (See Materials and Methods). Comparing this to the intervals indicated for the other materials in this length category, the confidence interval for Sanded Bestwall is clearly an underestimate; any intervals around frequencies from specific materials that are substantially smaller than others in the same length category also suggest other instances where variation may have been underestimated. Similar effects were observed on plots depicting width categories and plots depicting primary fibers and bundles (data not shown). The implications of such observations are considered further in the Discussion Section.

Using variances derived in the current study, ANOVA results indicate that the length distributions and, separately, the width distributions across the six materials are significantly different ($P \leq 0.01$ for tests involving primary structures and $P \leq 0.025$ for tests involving total structures). However, when 7RF3 and sanded ready mix are dropped from consideration, differences between the remaining four materials (sanded and unsanded Bestwall and sanded and unsanded dry mix) disappear ($P_{\text{adj}} \geq 0.23$ for tests involving primary structures and $P_{\text{adj}} \geq 0.21$ for tests involving total structures). Using variances derived from surrogate data, ANOVA results indicate no significant differences in material-specific fractions of fibers counted within each length category or in material-specific fractions of fibers counted within each width category ($P \geq 0.068$ for tests involving primary structures and $P \geq 0.13$ for tests involving total structures).

Comparison of the fraction of matrices across materials

Table 1 presents statistics indicating the fraction of primary structures that are matrices and the fraction of fibers observed to be components of matrices in these dusts. Also, presented in the table are the standard errors of the mean (SEMs) observed across the fractions in samples representing each material. As can be seen in the table, both the mean fraction of primary structures that are matrices and the mean fraction of fibers that are components of matrices are $>50\%$ for the five materials representing mixtures of chrysotile with calcium carbonate (and other minor components). In contrast, the mean fractions in the commercial fiber (7RF3) are only 18 and 23%, respectively.

Results of one-way ANOVA indicate that both the fraction of primary matrices and the fraction of fibers in matrices are significantly different across materials ($P < 0.00001$ in both cases). Excluding 7RF3, results are still significant ($P \leq 0.01$ in both cases). However, when both 7RF3 and sanded ready mix are excluded, there are no significant differences between Bestwall and dry mix, whether sanded or not ($P \geq 0.3$).

Note that, although surrogate data suitable for application to these categories of structures could not be found, the highly significant ($P < 0.00001$) differences found between 7RF3 and the other five materials suggests that these differences may very well persist, even if surrogate data had been applied.

To illustrate the nature of the matrices observed in these materials, Fig. 6 presents photomicrographs of several asbestos structures selected from those observed in both a sample of unsanded dry mix and a sample of settled dust generated by sanding (prepared and applied) ready mix. As can be seen in this figure, the dry mix itself and the sanded material both appear to be composed of particles and fibers, with many of the fibers partially embedded in (or at least associated with) non-asbestos particles.

**DISCUSSION**

As indicated above, differences between the fiber sizes of the six materials tested were only observed when the smaller variances between the samples in this study are used in the ANOVA. When variances derived from the surrogate data are used, the observed differences disappear. Thus, unless dust from commercial Grade 7 fiber exhibits substantially smaller temporal variation than that from commercial Grade 3 fiber (from which the surrogate data are derived), it is unlikely that differences between any of the materials studied here are greater than differences that would otherwise be observed between samples of any one material that are collected at different times (or, more to the point, created from samples of commercial fiber that were mined and milled at different times). This means that the fiber size distributions exhibited in dusts from these materials should not be considered to be different.

Even if commercial Grade 7 fiber exhibits somewhat less temporal variation than Grade 3 fiber (which would cause the true variances in the studied material to be somewhere in magnitude between that observed among samples in this study and that observed in the surrogate data), then (at most) we would expect to see only the differences reported above: (i) between the commercial fiber itself...
(7RF3) and the five other materials in which it is incorporated and (ii) between sanded reformulated joint compound and the dry mix joint compounds (Bestwall or re-created dry mix, whether sanded or not).

The implications of any real difference between 7RF3 (Fig. 4) and the five joint compound materials (observed when study-specific variance was used in the ANOVA) are interesting. It would suggest that when chrysotile fibers are mixed with calcium carbonate particles in dry mix (7RF3) and the five other materials in which it is incorporated and (ii) between sanded reformulated joint compound and the dry mix joint compounds (Bestwall or re-created dry mix, whether sanded or not).

Table 1. The fraction of structures represented by matrices

<table>
<thead>
<tr>
<th>Material</th>
<th>N</th>
<th>Fraction of primary structures that are matrices</th>
<th>Fraction of fibers and bundles found in matrices</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Mean</td>
<td>SD</td>
</tr>
<tr>
<td>Sanded Bestwall</td>
<td>2</td>
<td>0.56</td>
<td>0.092</td>
</tr>
<tr>
<td>Sanded dry mix</td>
<td>3</td>
<td>0.61</td>
<td>0.049</td>
</tr>
<tr>
<td>Sanded ready mix</td>
<td>3</td>
<td>0.80</td>
<td>0.017</td>
</tr>
<tr>
<td>Unsanded Bestwall</td>
<td>3</td>
<td>0.67</td>
<td>0.093</td>
</tr>
<tr>
<td>Unsanded dry mix</td>
<td>3</td>
<td>0.66</td>
<td>0.021</td>
</tr>
<tr>
<td>Unsanded 7RF3</td>
<td>3</td>
<td>0.18</td>
<td>0.053</td>
</tr>
</tbody>
</table>

*SEM, which equals the SD divided by the square root of the number of samples, N.
carbonate and other components of the joint compound, even as dry powders, the fibers, and carbonate particles (and/or the binder in the mix) tend to associate with one another. As a consequence, when dusts are generated from such mixtures, the fibers and either the carbonate or the binder tend to remain associated as matrices (particles containing chrysotile, carbonate, and binder), rather than becoming separated when disturbed, as they would if there was no force of attraction. Consistent with this hypothesis, the fractions of fibers associated with matrices in the dusts from the joint compounds are also significantly greater than those observed in the unmixed commercial fiber (7RF3) alone (Table 1). While one would expect this for sanded materials (because they would have been wetted and undergone reactions required to set-up the hardened lattice of an applied joint compound), it is surprising to observe the same frequency of matrices in unsanded materials, which are combinations of dry quantities of commercial fiber, calcium carbonate, and binders that are simply physically mixed.

If differences that were observed between the ready mix and both the re-created dry mix and the Bestwall are real, they may be attributable to a variety of factors, including (for example) differences in the behavior of the different types of binders used in the re-created dry mix and ready mix formulations (casein versus polyvinyl acetate, respectively, see Brorby et al., 2008) or natural variation in the source materials themselves. Thus, in addition to differences in size distributions (Fig. 4), both the fraction of matrices observed among primary structures and the fraction of fibers embedded in matrices (Table 1) in sanded ready mix dusts are somewhat greater than observed among the dry mixes. Importantly, however, given that at least the differences in size distributions disappear when variation from surrogate data are considered, it is not clear that these differences are meaningful or represent real differences between these materials. As previously indicated, no surrogate data were available for application to the data in Table 1.

Importantly, because the variances observed between samples in this study were almost uniformly smaller than those derived from the surrogate data, with the exception of comparisons involving sanding (discussed below), it is unlikely that increasing the number of samples in this study would result in detection of additional differences (facilitated by an increase in power). Instead, increased power would primarily increase the precision with which the irreducible variation of these materials can be characterized.

Comparing two materials whose properties are known to vary over time requires that the effects of such variation be explicitly considered as part of the comparison. Ideally, studies with this objective would best be conducted by collecting samples of each material that were manufactured over broadly varying time frames and looking for differences between the sets of samples that are larger than differences within the sets of samples. Lacking access to such diverse samples, however, surrogate data were employed to provide at least a rough estimate of the degree of variation that would likely be observed among more representative sets of samples.

As indicated above, the re-created dry mix and the historically manufactured Bestwall (whether sanded or not) were not found to be different from one another (even when the smaller study-specific variances were employed in the ANOVA). This suggests that the properties of the dry mix re-created for this study reasonably reflect the properties of the historical dry mix (Bestwall).

In contrast to the above, when the objective of a study is to compare the effects of an application (such as sanding) on a material, it is best to explicitly compare ‘before’ and ‘after’ samples that are homogenized splits of the same starting material (as was done in this study); one wants to remove the effects of irreducible variation within the material being sanded so that the effects of sanding itself can be more easily detected. Consequently, that no differences between sanded and unsanded Bestwall or between sanded and unsanded dry mix were detected, even when the smaller variances from the current study were used in the ANOVA, suggests that the effects of sanding are small. Nevertheless, it is possible that increasing the number of samples in this kind of study would change this result. However, given that the irreducible variation within materials is apparently larger than any differences that the effects of sanding might produce, it is reasonable to conclude that, even if such differences were to be detected, they are unlikely to be of practical importance.

That the effects of sanding are small suggests that the act of sanding primarily tends to disaggregate the matrix that forms when calcium carbonate and binder either react with water or lose water (during mixing, application, and drying) rather than actually breaking the fibers themselves. Such findings are consistent with findings reported elsewhere indicating that the characteristics of dusts generated from mechanical processes tend to be mediated more by the properties of the bulk materials from which the dusts derive than by the nature of the mechanical processes causing the dust to be generated (Wylie
and Schweitzer, 1982; Turcotte, 1986; Wylie, 1993; reviewed in Berman, 2010a).

Interestingly, recent studies of dusts generated from sanding of (asbestos-free) paints, coatings, and cements with and without various types of nanoparticle additives (e.g. Koponen et al., 2009, 2011; Göhler et al., 2010; Wohlleben et al., 2011) suggest that mechanical processes play a more important role in mediating dust characteristics than suggested in this study. Comparisons must be made carefully, however, due to differences in study focus, experimental design, and analytical approaches. Regarding analytical approaches, for example, these other studies derive size distributions of non-fibrous particles by integrating signals from indirect detection methods. In contrast, fibers are directly observed under a microscope and manually counted and sized in the current study (while other non-fibrous particles are ignored). Thus, the particle populations studied are very different; while this study focuses on what are primarily single-crystal fibers and aggregates of such fibers, the others focus on undifferentiated populations that may include single crystals and composite particles composed of multiple types of materials.

Inferences from the other studies cited above must also be viewed with caution, given the limitations of each study’s design. In three studies (Koponen et al., 2009, 2011; Göhler et al. 2010), for example, all comparisons were between dusts generated by sanding of different materials but under identical conditions. Thus, any inferences stated about the effects of mechanical processing were necessarily based on the introductory reviews of other studies rather than findings from the studies themselves. Moreover, based on the reviews presented, such inferences seem equivocal at best.

One finding is consistent across all four studies of non-asbestos materials; none report observation in dust of isolated nanoparticles free of the surrounding matrix in which they are embedded. This is consistent with the observation from the current study that joint-compound dusts contain increased fractions of matrix particles relative to dusts from milled fiber (whether sanded or not) and it suggests that sanding (whether mechanical as employed in the other studies or manual as employed in this study) does not impart sufficient energy to fully disaggregate sanded composites into their components (whether paints, coatings, cements, or joint compounds). Thus, particle aggregates appear to survive the sanding process and their size may be determined by the mechanical energy applied; increasing energy will lead to increasing disaggregation. Importantly, none of the findings from the four studies are inconsistent with the theoretical basis articulated in the introduction to support use of the MEM for reconstructing historical asbestos exposures.

CONCLUSIONS

Findings from this study indicate that (i) chrysotile-containing joint compounds can be re-created using historical formulations, such that the characteristics of the modern material reasonably mimic those of a corresponding historical material; (ii) dusts generated from joint compounds are different from dusts generated from commercial chrysotile fiber, in that a majority of fibers in joint compound dusts (whether sanded or not) are associated with matrices; and (iii) the effects of sanding on fiber size distributions are small so that sanding serves primarily to disaggregate the hardened matrix of an applied material rather than breaking fibers.

FUNDING

This research was funded by Georgia-Pacific LLC, which has been in litigation related to joint compound.

APPENDIX 1

Table A1 and A2, respectively, present the fraction of primary fibers and bundles and total fibers and bundles observed in each of 15 size categories among the six materials characterized in this study (along with the SEM for each fraction estimate).

Acknowledgements—The authors wish to thank Tony Kolk (EMS Laboratories, Pasadena, CA, USA) for his input and suggestions during sample preparation and analysis and R. J. Lee (Monroeville, PA, USA) for their assistance providing photomicrographs. We would also like to acknowledge input of the anonymous peer-reviewers, whose comments and suggestions helped us to strengthen and simplify this manuscript. All the authors are involved in asbestos consulting in one form or another. Stewart E. Holm has recently given testimony in joint compound litigation.

REFERENCES


Goehler from sanding of (asbestos-free) paints, coatings, and Schweitzer, 1982; Turcotte, 1986; Wylie, 1993; also be viewed with caution, given the limitations of crystals and composite particles composed of differentiated populations that may include single aggregates of such fibers, the others focus on un-on what are primarily single-crystal fibers and a microscope and manually counted and sized in by integrating signals from indirect detection meth-analytical approaches, for example, these other stud-haviors helped us to strengthen and simplify this manuscript. Thus, any inferences stated about the effects of sanding on fiber size distributions are increasing disaggregation. Importantly, none of the cle aggregates appear to survive the sanding process (whether mechanical as employed in the other stud- (whether sanded or not) and it suggests that sanding (fallen into place? Crit Rev Toxicol; 40: 151–88. One finding is consistent across all four studies of TABLE A1 and A2, respectively, present the fraction (along with the SEM for each fraction estimate). Berman DW. (2010b) Response to “Letter concerning “Comparing milled fiber, Quebec ore, and textile factory dust: has an-other piece of the asbestos puzzle fallen into place?” by Berman” by Dement and Synder. Crit Rev Toxicol; 40: 752–7. Berman DW. (2011) Apples to apples: the origin and magni-tude of differences in asbestos cancer risk estimates derived using varying protocols. Risk Anal; DOI: 10.1111/j.1539-6924.2010.01581.x.


Table A1. Fraction of primary fibers and bundles in indicated size categories and their associated standard errors.

<table>
<thead>
<tr>
<th>Activity material</th>
<th>Size (µm): L ≤ 5</th>
<th>5 ≤ L &lt; 10</th>
<th>10 ≤ L &lt; 20</th>
<th>20 ≤ L &lt; 40</th>
<th>40 ≤ L</th>
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<tbody>
<tr>
<td></td>
<td>0.25 &lt; W ≤ 0.4</td>
<td>0.25 &lt; W ≤ 0.4</td>
<td>0.25 &lt; W ≤ 0.4</td>
<td>0.25 &lt; W ≤ 0.4</td>
<td>0.25 &lt; W ≤ 0.4</td>
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<td>Sanded BestWall</td>
<td>0.7754 0.0254 0.1017 0.0144 0.0321 0.0059 0.0041 0.0017 0.0023 0.0087 0.0000 0.0007 0.0014</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sanded dry mix</td>
<td>0.7831 0.0344 0.0174 0.0146 0.0064 0.0055 0.0056 0.0009 0.0019 0.0054 0.0001 0.0004 0.0006</td>
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<td></td>
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<tr>
<td>Sanded ready mix</td>
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<td></td>
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<td></td>
</tr>
<tr>
<td>Unsanded BestWall</td>
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Fraction of total in each size category

<table>
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<th>Activity material</th>
<th>Sanded BestWall</th>
<th>Sanded dry mix</th>
<th>Sanded ready mix</th>
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<th>Unsanded C7RF3</th>
<th>Unsanded dry mix</th>
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<td>Sanded dry mix</td>
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<td>Sanded ready mix</td>
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<td>Unsanded BestWall</td>
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<tr>
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</tbody>
</table>

Standard error of the mean (SEM) for each size category

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<th>Sanded ready mix</th>
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<th>Unsanded C7RF3</th>
<th>Unsanded dry mix</th>
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</tr>
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<td></td>
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</tr>
<tr>
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<td>0.0163 0.0136 0.0128 0.0023 0.0028 0.0029 0.0031 0.0019 0.0010 0.0016 0.0001 0.0002 0.0002</td>
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<tr>
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Table A2. Fraction of total fibers and bundles in indicated size categories and their associated standard errors.

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<th>Activity material</th>
<th>Size (μm): L ≤ 5</th>
<th>5 ≤ L &lt; 10</th>
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<td>0.0105</td>
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<td>0.7579</td>
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<td>Unsanded dry mix</td>
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<td>0.0253</td>
<td>0.0347</td>
<td>0.0237</td>
<td>0.0020</td>
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</table>

Fraction of total in each size category

<table>
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<tr>
<th>Activity material</th>
<th>Sanded BestWall</th>
<th>Sanded dry mix</th>
<th>Sanded ready mix</th>
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<th>Unsanded C7RF3</th>
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<tr>
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<td>0.0066</td>
<td>0.0370</td>
<td>0.0012</td>
<td>0.0010</td>
<td>0.0042</td>
</tr>
<tr>
<td>Sanded dry mix</td>
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<td>0.0088</td>
<td>0.0046</td>
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<td>0.0026</td>
<td>0.0034</td>
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<td>0.0011</td>
<td>0.0007</td>
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<td>0.0023</td>
<td>0.0028</td>
<td>0.0019</td>
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<td>0.0037</td>
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SEM for each size category

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<td>0.0011</td>
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<tr>
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<td>Unsanded C7RF3</td>
<td>0.0779</td>
<td>0.0235</td>
<td>0.0324</td>
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<td>0.0036</td>
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<td>0.0008</td>
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</table>