Comparative HRTEM Examination of Carbon Black and Soot Structures

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ABSTRACT

Soot is a major source of airborne particulate that forms as an undesired combustion byproduct from a variety of sources, such as wildfired, candles, and diesel exhaust, whereas carbon black is a controlled combustion product with specific properties designed for various applications. An "aciniform" structure is a common characteristic of soot and carbon black, and a method currently exists to compare carbon black to various soot sources and other darkening agents. High resolution transmission electron microscopy [HRTEM] characterization of such aciniform carbon particles has been reported previously, but no comparison of microstructural variation in aciniform carbon primary particles from multiple sources is publicly available. The authors have investigated aciniform primary particles from varaious sources using HRTEM, with the ultimate goal of developing an atlas of microstructural and chemical characteristics to aid in identifying the sources of soot collected from ambient air and settled dust.

Keywords: carbon black, soot, HRTEM

1 INTRODUCTION

Aciniform carbon structures are a subject of interest in health and safety, in the case of both soot and carbon black particulate[1,2], as well as in industrial applications of carbon black. The ASTM Method, "Standard Practice for Sampling and Testing of Possible Carbon Black Fugitive Emissions or Other Environmental Particulate, or Both," provides a guideline for comparing carbon black to various soot sources and darkening agents[3]. HRTEM characterization of soot microstructures have been reported previously[4], with diesel soot representing a particulate of special interest[5,6]. A greater understanding of microstructural variation of soot and carbon black primary particles would compliment existing research on potential hazards and applications of soot and carbon black particulate. Currently, no such investigation of primary particle microstructural variation of soot and carbon black exists, and this investigation is the beginning of an analysis of microstructural variations, with the end goal of establishing an archive of microstructural and chemical properties of aciniform carbon structures, for the purpose of aiding individuals interested in determining sources for aciniform carbon particulate observed and collected in ambient air or settled dust samples.

2 MATERIALS AND METHODS

Seven varieties of aciniform carbon were prepared for microstructure characterization (Table 1). HRTEM grid preparation varied depending on the initial condition of the sample. Samples that originated from settled dust using a collected wipe were prepared by cutting a section, a maximum of 0.5cm X 3cm in area, from the wipe. The wipe sections were then placed into 1-dram vials pre-rinsed with spectranalyzed acetone. Spectranalyzed acetone was added to the vial and strip, and the vials were ultrasonicated for 10 minutes to suspend the wipe particulate in the spectranalyzed acetone. Subsamples of material that originated from a bulk container were transferred with a spatula to 1-dram vials pre-rinsed with spectranalyzed acetone. Spectranalyzed acetone was then added to the vials, and the vials were ultrasonicated for 10 minutes in order to suspend the bulk material subsample in the spectranalyzed acetone.

Suspensions were deposited directly onto 200 mesh carbon-coated copper TEM grids using a 10 microliter pipette. All samples collected for this study were analyzed using a Philips CM200 HRTEM operated with a LaB_6 filament at an accelerating voltage of 180kV.

Sample	Description
Wildfire Soot	Alcohol wipe of settled dust on hard interior surfaces in a building proximal to a California wildfire
Candle Soot	Microvac sampler proximal to source; 0.45µm pore size mixed cellulose ester filter (sample prepared as a wipe)
Front End Loader Soot	Tailpipe wipe sample from a diesel front end loader
Truck Diesel Soot	Cotton swab sample of tailpipe from Ford F-250 V8 diesel truck (sample prepared as a wipe)
NIST SRM 2975 Diesel Soot	Bulk NIST SRM 2975 industrial forklift diesel particulate matter
Carbon Black #1	Bulk N990 carbon black reference
Carbon Black #2	Bulk N326 carbon black reference

Table 1. List of aciniform carbon varieties prepared for microstructure characterization.

3 RESULTS AND DISCUSSION

Representative photomicrographs obtained during the examination of each sample are provided in Figures 1 through 14. Lower magnification (TEM) images were obtained at a nominal magnification ranging between 5,000 to 25,000 times. Higher magnification (HRTEM) images were obtained at a nominal magnification ranging between 300,000 to 410,000 times. The lower magnification images reveal variations in overall aggregate structures. For example, wildfire (Figure 1) and candle soot (Figure 3) show great variation in primary particle size distributions within any individual aggregate whereas diesel soot (Figures 5, 7, and 9) and carbon black (Figures 11 and 13) are more uniform in primary particle size.

The HRTEM images reveal variations in primary particle structure. Soot from uncontrolled sources, like the wildfire soot (Figure 2), show irregular edges for individual primary particles Candle soot and diesel soot primary particles are more well-structured (Figures 4, 6, 8 and 10); however, these also typically have irregular edges and poorly resolved microstructure (turbostratic layers). Only the carbon black samples showed both well defined primary particle edges as well as microstructure (Figures 12 and 14).

These findings support known characteristics of carbon black in literature and with the carbon black characteristics specified in the ASTM D6602 standard test method; however, additional work to characterize more samples would further broaden our understanding of microstructural variations and chemical compositions from different sources of soot and carbon black.

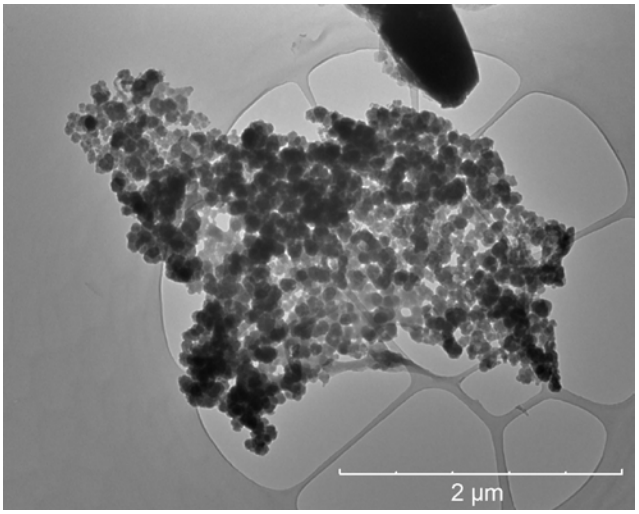

Figure 1. California wildfire soot.

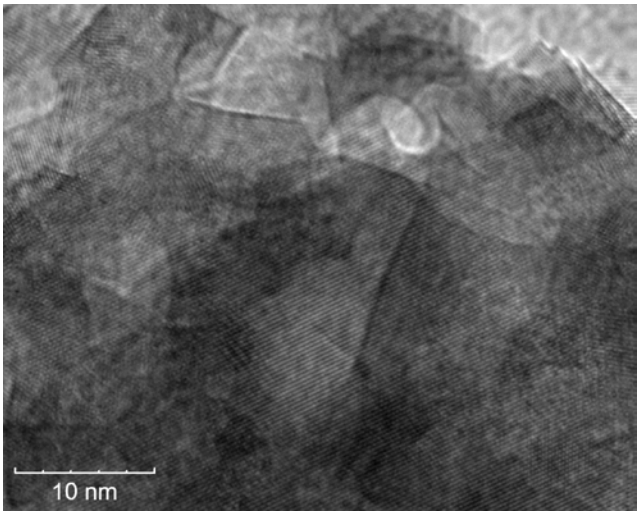

Figure 2. California wildfire soot.

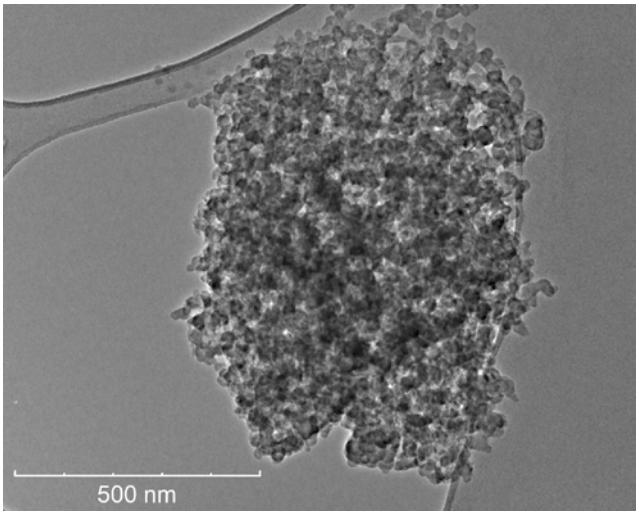

Figure 3. Candle soot.

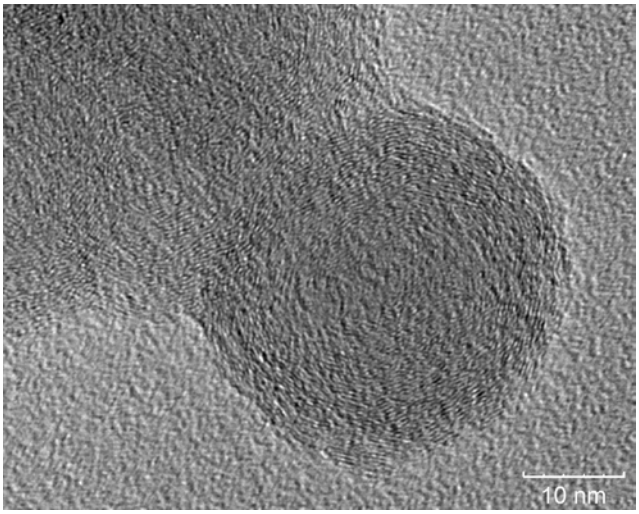

Figure 4. Candle soot.

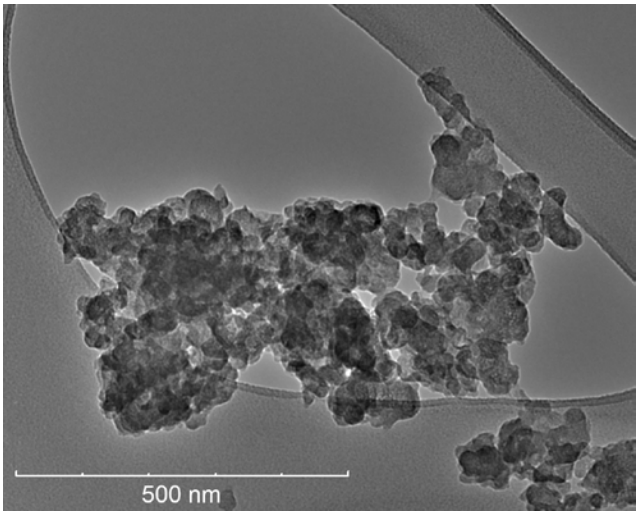

Figure 5. Front end loader diesel soot.

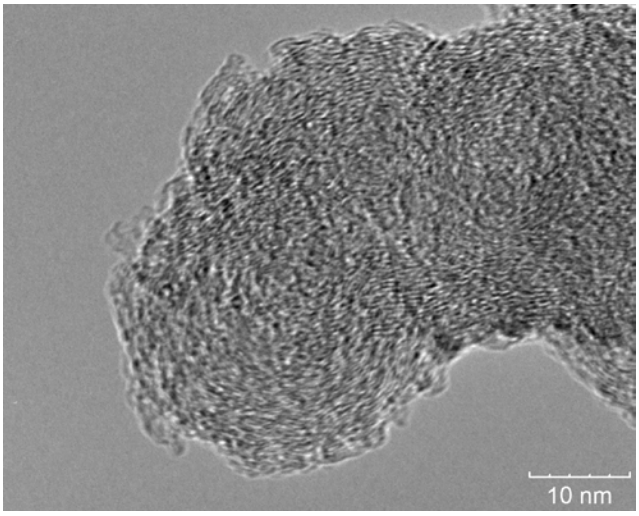

Figure 6. Front end loader diesel soot.

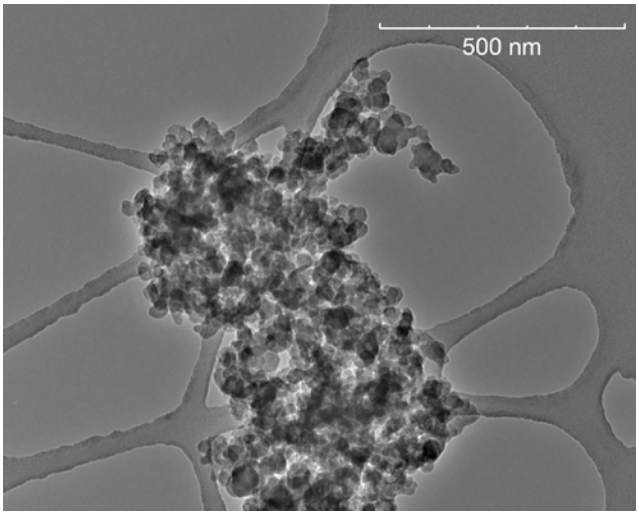

Figure 7. Truck tailpipe diesel soot.

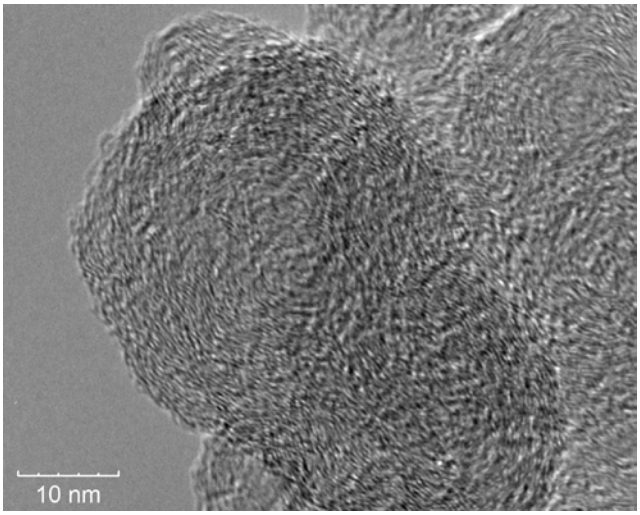

Figure 8. Truck tailpipe diesel soot.

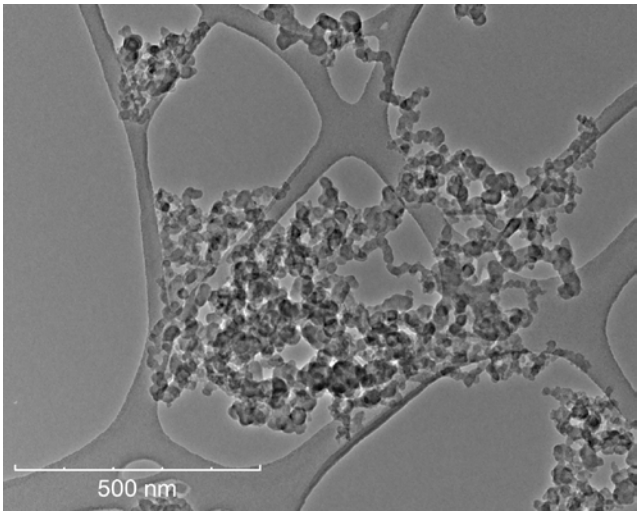

Figure 9. NIST SRM 2975 diesel soot.

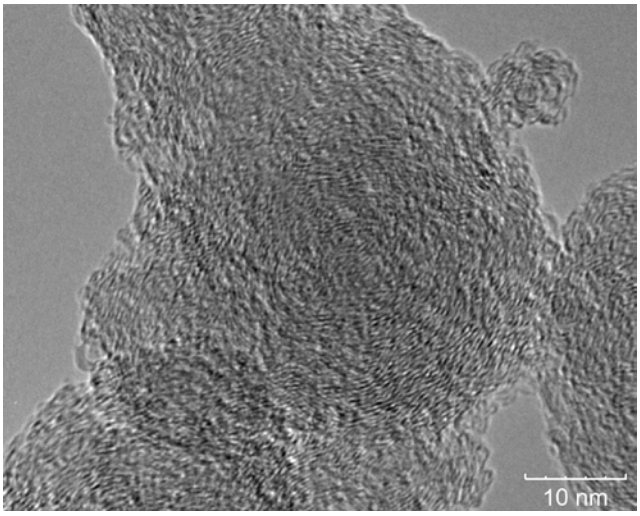

Figure 10. NIST SRM 2975 diesel soot.

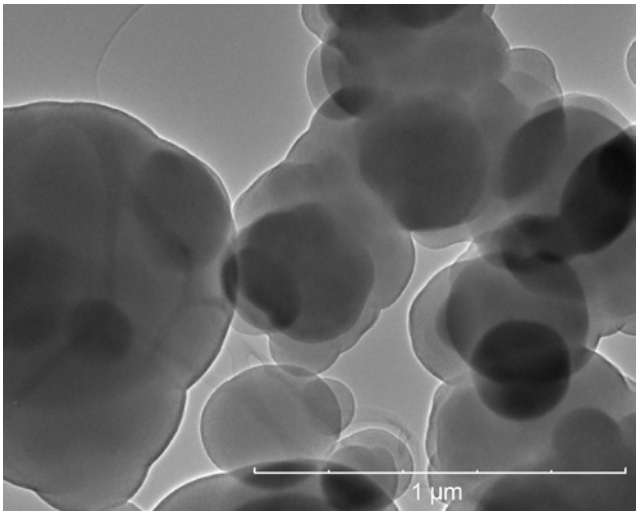

Figure 11. N990 carbon black reference.

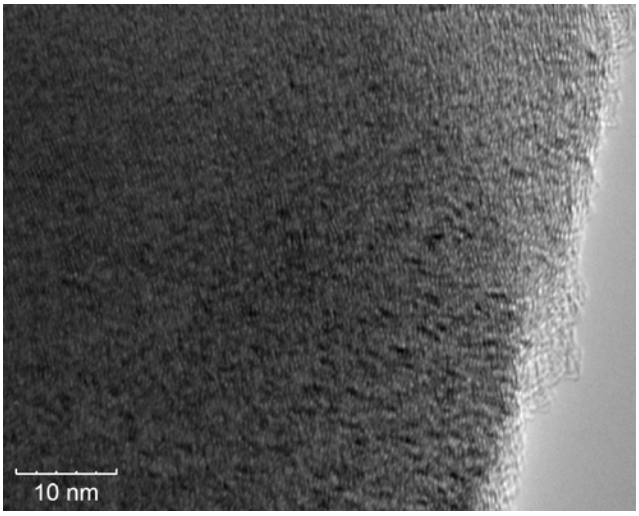

Figure 12. N990 carbon black reference.

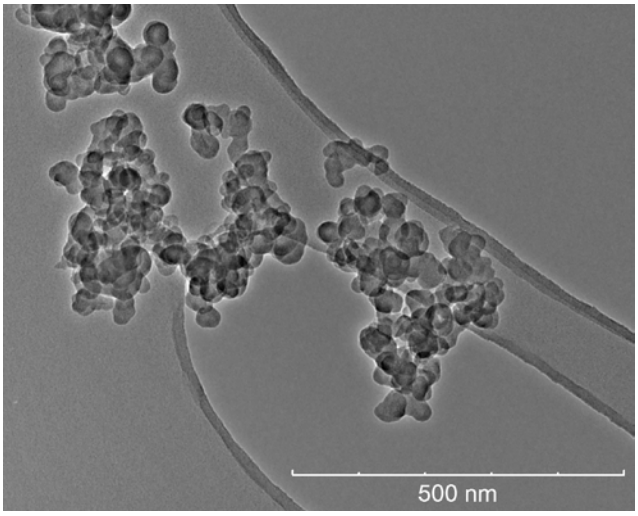

Figure 13. N326 carbon black reference.

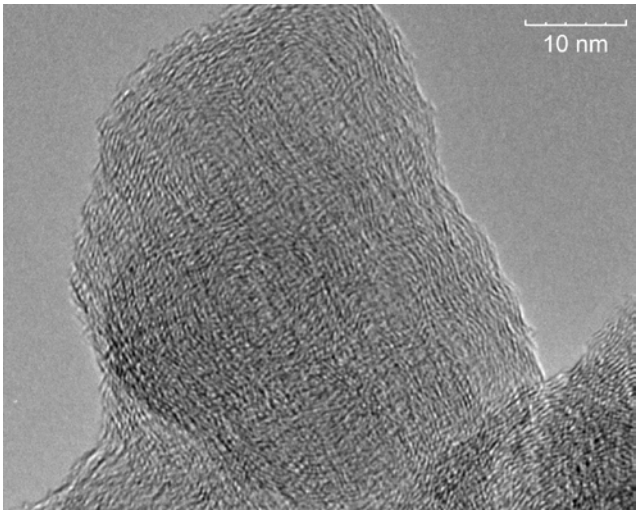

Figure 14. N326 carbon black reference.

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