A New Approach to Understanding the Mechanism and Effect of Phase Change of Aluminum in Aluminum Nanoparticles Oxidation: An Experimental Study

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Abstract— Recently, Aluminum nanoparticles become more and more interesting due to their possible applications in explosive materials. The current study aims to investigate, qualitatively, the oxidation of aluminum nanoparticles with passivating oxide coating. The stability of the oxide coating in nanoaluminum was evaluated by hot-stage transmission electron microscopy (TEM). In addition, single particle mass spectrometer (SPMS) was used to assess the oxidation process. The results show that the oxidation of oxide-coated nanoaluminum not only happens, simultaneously, with melting of the aluminum core, but also it is most probably that it begins with that event and resulted mechanical rupture of the oxide coating.

Index Terms— Mechanism, Phase change, Aluminium, Nanoparticles, Oxidation, Nanoaluminium, Oxide coating, Hot-stage transmission electron microscopy (TEM), Particle mass spectrometer (SPMS)

1 Introduction

The energetic materials are defined as the materials with high capacity of chemical energy storage and high rate of energy release [1-8]. These materials are of numerous applications in explosives, propellants and, pyrotechnics [9, 10]. For example, micro-aluminum, with a high enthalpy of combustion, is usually used in rocket propellant formulations [11-25]. There are various studies in the literature about the combustion mechanism of micro-aluminum particles [26-31]. It was implied by some studies [32, 33] that the ignition temperature of aluminum oxide is coinciding with its melting point [34-39]. However, with application of embedded thermocouples, other researchers [40] were found that aluminum is ignited at temperature of 2000-2100 K [41]. Moreover, there are some studies about the temperature of fracture in oxide shell which report this temperature, and hence, ignition, could be as low as 1300 K [42].

The above mentioned studies are all about micro-particles [43]. However, fine grained aluminum (nanoaluminum) is recently more interested as the grained metals are well-known for their highly reactivity [44-56]. As there will be a direct relationship between the transfer of oxidizer to the particle and the rate of energy release, it may be concluded that smaller particles will accelerate the overall energy release [57, 58].

Consecutively, this question is raised that what is the difference between burning properties of nanoaluminum and micro-aluminum particles [59]. The applicability of aluminum nanoparticles in increasing of the burning rate of propellants by 5-10 times compared to usual aluminum particles is recently shown by [60]. Furthermore, it was reported that the ignition temperature of aluminum nanoparticles formed by electrical wire explosion may be low to 820 K, as measured by thermogravimetry (TGA) and differential thermal analysis (DTA) [61]. It should be noted that conventional dynamic thermal techniques such as TGA which need to a bulk sample are used in most of recent measurements [62]. Subsequently, it also should be noted that the response dependency of these methods to heat and mass transfer effects, which are sorely explainable, is well-known [63-73]. Ideally, a basic analysis of aluminum nanoparticle oxidation is performed on a single particle without any consideration of heat and mass transfer effects between the considered particle and its surrounding particles which interacts with this [74, 75].

The current study aims to investigate, qualitatively, the basic reactivity of aluminum nanoparticles and to show the importance of aluminum phase change in the oxidation process and the role and stability of the oxide coating. Oxide has an important role since it can be a passivating layer for bare and fine metal particles which can be pyrophoric [76-84]. In the current study, the results of using the hot-stage transmission electron microscopy (HSTEM) and single particle mass spectrometer (SPMS) [85] for characterization of the nanoaluminum oxidation were reported.

2 Experimental Methods

Commercially available aluminum nanoparticles (Aveka, corp.) that dispersed in methanol were used in this study. The results of TEM analysis was shown that the particles were aggregates of around 170 nm, and mainly composed of particles between 40-60 nm, with a passivating oxide coating lower than 15 nm.

The marked silicon oxide coated nickel grids were used in the microscopy experiments. The methanol dispersion was ultrasonicated before the deposition of the mixture to the grid. The heating and continuous monitoring of particles on a hot-stage TEM (Philips CM30) to a temperature of 1195 K, under vacuum, was performed and to clear the role of thermal

stresses on the particles. In the next part of the experimental study, a center marker was used to locate several particles on the grid. Then, the grid was removed from the microscope and heated in a tube furnace in the presence of air at various temperatures. Finally, the grid was returned to the microscope and the same particles were located using the marker to detect the morphological changes.

Another experiment also was performed in which a newly developed single particle mass-spectrometer (SPMS) [85] with the ability of quantitative determination of the relative elemental composition of individual nanoparticles was used to measure the temperature of oxidation initiating in heated air. Dry compressed air was used in these experiments for aerosolizing of the aluminum nanoparticle/methanol dispersion. The removal of methanol from the aerosol stream was performed by passing the stream through several diffusion dryers. Ultimately, the stream was heated to different temperatures, and SPMS system was used, based on a procedure described in [85], to analyze the particles in the stream.

3 Results and Discussions

The oxide shell, as estimated to be about 15 nm, in intact but it is clearly cracked and open with an obvious meniscus which it can be ascribed to the withdrawing liquid aluminum interface as it flows out of the particles. Based on the bulk properties as a rough estimate, it should be noted that the density of liquid aluminum (9.6 g/cm3) is less than that of solid aluminum (9.9 g/cm3), since aluminum expands by 18% as it melts. By neglecting the thermal expansion of the oxide shell, which is reasonable at these temperatures relative to the expected changes for aluminum, the oxide shell will be under tension and the aluminum core under compression. Assuming that the bulk modulus of aluminum (88 Gpa) is useable at these length scales, a 93000 atm rise in internal pressure will be present at the oxide shell because of the density difference. The presence of a large internal pressure on the aluminum core and tension on the oxide shell at these high temperatures is demonstrated, recently, by a molecular dynamic calculation [83]. These mean that the oxide shell is dynamically unstable upon melting of the aluminum core. In addition, the results are shown that the increase in pressure in smaller particles is higher than that in larger particles, demonstrating that smaller particles are of higher tendency to rupture. Furthermore, the oxide coating in smaller particles is under higher tension than large particles due to its higher curvature which in turn leads to more easy rupture in smaller particles.

In all cases, particles were heated in air for 20 minutes. It is seeable that the before and after images at 914 K are basically identical, while particles heated to a temperature of 1212 K, above the melting point (952 K) of aluminum, clearly show considerable restructuring and rupture/loss of apparent oxide layer structure. Although these results are consistent with the hot-stage TEM measurements, they still deal only with the issue of physical restructuring and melting of aluminum and do not address the issue of any chemical change, i.e., oxidation.

The single particle mass spectrometer (SPMS) was used to investigate the oxidation as opposed to melting and to track the oxygen content of particles under exposure to air at different temperatures. In these experiments, aluminum aerosol resides about one second in the heated section of the flow reactor.

It can be seen that the oxygen has not appeared in the spectra until a temperature of 992 K, which is slightly above the melting point of aluminum, while at 108 K lower, any oxidation is not observed. With increasing temperature the oxygen signal intensifies, implying greater extent of oxidation. A sensitivity of about 1.5% (mole percentage) observed in the particle in previous work with the SPMS for mixed composition or coated particles. With 13-14 nm oxide thickness, the SPMS should detect the oxide if it exists. But in these experiments, where aluminum dispersed in methanol was used unlike the particles used in microscopy experiments, there is not an initial oxide coating since they were never exposed to ambient atmosphere. In our experimental conditions, the air was mixed with aluminum particles just before the furnace and the exposed time for aluminum particles to the air was relatively short (1.5 s). Clearly, such a time is too short and hence, the reactivity of nanoaluminum is sufficiently slow as to prevent the formation of a detectable oxide coating. At a temperature of 992 K, the oxygen is appeared in the particle, as consistent with the above mentioned microscopy studies. The current work is aimed to quantifying the kinetics of oxidation using this experimental approach.

Based on the results of these two different experiments, it is understand that there is a mechanism whereby the mechanical stability of the oxide shell determines the onset of combustion. With increasing of the temperature beyond the melting point, the density difference between aluminum solid and liquid causes a rupture in the oxide shell. This, in turn, results in exposure of aluminum to the oxidizer and following ignition.

Previously, research groups [84] were reported that the oxidation of nanoaluminum is happened at temperatures of around 800 K, which is below the melting point of aluminum. This is significantly different from observations of the current study. These researchers used dynamic thermal techniques such as thermogravimetry. Although these techniques are widely used to measure condensed phase reactions, limitations of these techniques are well-known due to uncertainties related to heat and mass transfer [83-85]. The use of both these methods, SPMS and TGA, in kinetic measurements of solid-state reactions resulted that, for example, the onset temperature of thermal decomposition reactions of metal nitrates varied with the variation of mass loading in the case of TGA [83-85]. It was reported that the onset temperature was consistently lower with TGA, and that small samples tended to increase the onset temperature [83-85]. The difference is that the conventional methods use sample sizes of the order of a milligram, while the SPMS measurement characterizes a single nanoparticle, of the order of a femtogram. It has been shown that increase in sample size led to decrease in activation energy of a reaction whivh this in turn led to measuring a lower onset temperature than that measured by conventional methods. It may be due to the fact that higher sample size would result in heat release which raises the temperature of the sam-

ple above the measured pan temperature, and therefore an observation of an apparent higher reactivity. The authors believed that a sample size of approximately 1.5 fg will be mitigates some of these issues and makes a more direct and clear observation of the onset condition to oxidation.

4 Conclusion

The current study aims to evaluate the role of the mechanical stability of the oxide shell over an aluminum nanoparticle and its role in passivating the particle toward oxidation. The hot-stage TEM imaging and single particle mass spectrometry were used to reveal the morphological and chemical changes, respectively. Based on the results of both experimental studies, it was concluded that aluminum phase change causes rupture of the oxide shell, and may be the primary initiator in the ignition of aluminum nanoparticles. It may be interesting for who are deals with new propellant formulations based on metal nanoparticles.

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