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PHYSICOCHEMICAL PROCESSES IN ALUMINUM NANOPARTICLES AT HIGH TEMPERATURES

A.S. Abiyev[1,2]

[1]National Nuclear Research Center, Baku, Azerbaijan
[2]Joint Institute for Nuclear Research, Dubna, Russia

Abstract. The physicochemical properties of Al nanoparticles have been studied at high temperatures. In this work, d = 40-60 nm nanoparticles with a cubic crystal structure were studied. The study of thermophysical properties was carried out on a Differential Scanning Calorimeter (DSC) in the temperature range T = 30-700 °C. It has been established that thermal transitions are observed at T = 278 °C and T = 616 °C. The effect at T = 278 °C was explained by the decomposition of hydroxide groups, and the effect at T = 616 °C was explained by the melting process.

Keywords: *nanoparticles, Al, DSC, thermal properties.*

Corresponding Author: *A.S. Abiyev, Department of Nanotechnology and Radiation Material Science, National Nuclear Research Center, AZ-1073, Baku, Azerbaijan, e-mail: afsunabiyev@gmail.com*

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1. Introduction

Observations of new effects on nanoscale materials further increased interest in these materials. It is known that as particle size decreases, their functionality increases. Therefore, the study of the physicochemical properties of nanomaterials of different sizes expands the scope of these materials. Nanoparticles of metals and metal oxides are also of great interest, since they are used in the synthesis of composite materials (Qu *et al.*, 2007; Rzayev *et al.*, 2021; Beril Tuğrul *et al.*, 2021; Demir *et al.*, 2018; Ali *et al.*, 2021).

It is known that many physical properties of materials depend on their size. Therefore, the study of size effects in materials helps to explain many of the processes occurring in them. It has been established that the ionic radii of elements can also affect the formation of crystallites in some compounds (Jabarov *et al.*, 2014). As can be seen, not only the electrical properties, also the structural properties can change depending on the type of ions that make up the materials.

The study of thermal properties at high temperatures helps to obtain additional information about their structural aspects. It has been found that it is possible to study chemical processes such as decomposition, oxidation and melting in compounds using a Differential Scanning Calorimeter (DSC). This method makes it possible to study not only the change in the state of aggregation, but also in general the phase transitions occurring in these materials. To minimize the oxidation of materials, studies are carried out in an inert gas environment. However, it has been found that an additional oxide layer can form on the surface of both non-oxides and complex oxides. The DSC method also makes it possible to calculate the thickness of this layer (Agayev *et al.*, 2020a, 2020b).

One of the most widely studied nanomaterials are aluminum nanoparticles. It is known that Al is widely used in various fields. The study of nanoparticles of this metal will serve to reduce the size of its areas of application. Structural studies have shown that Al nanoparticles combine with water molecules to form $Al(HO)_3$. Therefore, the structure of Al nanoparticles in the open air consists of two phases.mThe first phase corresponds to the cubic symmety crystal structure of aluminum with space group Fm-3m. The crystal structure of the second phase corresponds to the monoclinic symmetry crystal structure of the $Al(HO)_3$ compound with space group P_12_1/a_1. It has been established that the irradiation of these samples with high-intensity electron beams results in the destruction of hydroxide groups and the formation of single-phase pure Al nanoparticles (Jabarov *et al*., 2021; Abdullayeva *et al*., 2020).

Although the crystal structure and atomic dynamics of Al nanoparticles have been studied, their thermal properties have not been studied. It is known that the physical properties of materials depend on temperature. Therefore, it is very important to study the physicochemical processes occurring in materials under the influence of temperature. In this work, we studied the thermal properties of Al nanoparticles with a size of d = 40–60 nm. The studies were carried out by DSC in the temperature range T = 30-700 °C.

2. Experimental part

Nanoscale Al particles were used as received and were bought from SkySpring Nanomaterials, Inc. (SkySpring Nanomaterials, Inc. 2935 Westhollow Drive, Houston, TX, 77082, USA). Purity (99.9%) and size range (40–60 nm) of these particles has already been reported (https://www.ssnano.com/).

The crystal structure and phase analysis of the studied samples were carried out by the X-ray diffraction method. An Advance 8 diffractometer (Bruker, Germany) was used in the work. The parameters of this diffractometer were: 40 kV, 40 mA, CuKα - radiation, λ = 1.5406 Å. The X-ray diffraction spectra obtained at room temperature and under normal conditions were analyzed by the Rietveld method. The analysis was carried out in the Fullprof program.

The DSC studies were carried out using the STA 6000 simultaneous Thermal Analyzer which is manufactured by Perkin Elmer, USA. The standard adiabatic calorimetry was performed in the temperature range of 30-700 ºC at a heating rate of 5 ºC/min under the argon atmosphere at the flow rate of 20 ml/min. The cooling process was achieved with the help of the PolyScience analyzer cooling system and “digital temperature controller (Azimova *et al.*, 2020; Jabarov *et al*., 2021).

3. Results and discussions

The crystal structure of Al nanoparticles was studied by X-ray diffraction at room temperature. The X-ray diffraction spectrum obtained in the range of diffraction angles $10° \geq 2\theta \geq 90°$ is shown in Fig. 1. Structural studies have shown that Al nanoparticles combine with water molecules to form $Al(HO)_3$. The first phase corresponds to the cubic symmetry crystal structure of aluminum with space group Fm-3m. The unit cell parameters for this phase correspond to: $a = b = c$ = 4.054(8) Å. The crystal structure of the second phase corresponds to the monoclinic crystal structure of the $Al(HO)_3$ compound with space group P_12_1/a_1. The lattice parameters of this phase correspond to:

a = 5.051(9) Å, *b* = 8.679(6) Å, *c* = 4.792(4) Å and *β* = 91.(1)°. As can be seen, Al nanoparticles combine with water molecules in the air, forming $Al(HO)_3$. Therefore, these materials should be stored indoors. Possibly, small amounts of AlO or Al_2O_3 compounds are obtained in this compound. However, they were not observed in the X-ray diffraction spectrum. It is known that as the dimensions of materials change, so does their functionality. They are more active in smaller sizes. Active particles combine faster with oxygen and water molecules. When using nanoparticles in composite materials, the synthesis processes must be carried out either in a vacuum or in an inert gas medium.

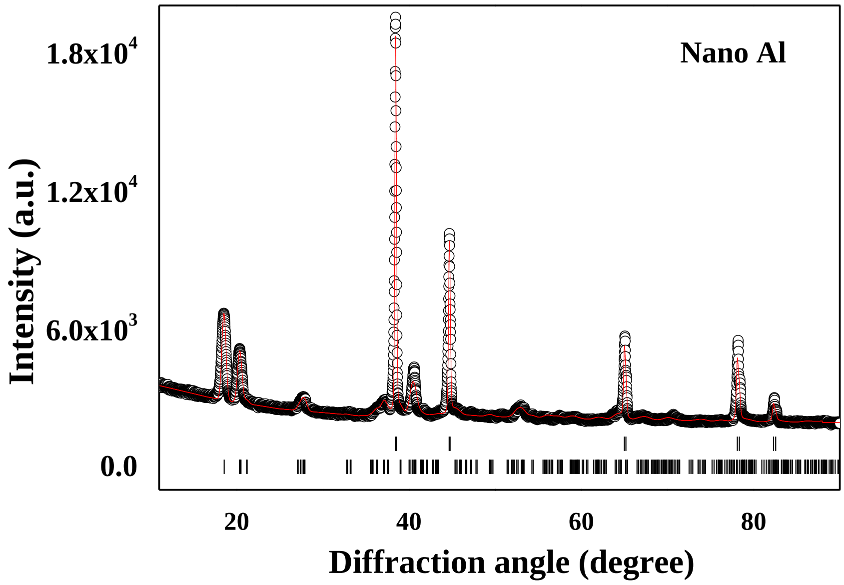

Fig. 1. X-ray diffraction spectrum of Al nanoparticles

In this work, we studied the thermophysical properties of Al nanoparticles. Structural studies have shown that aluminum nanoparticles combine with water molecules to form hydroxide groups. Thermal analysis studies are needed to observe these effects. The spectrum of the differential scanning calorimeter obtained in the high-temperature region is shown in Fig. 1.

The DSC spectrum obtained in the temperature range *T* = 30-700 °C was analyzed using the Origin program. It is established that the temperature dependence of the heat flux function consists of 5 intervals and 2 main effects. These intervals are:

1. *T* = 30-223 °C – constant region of the heat flux function,
2. *T* = 223-306 °C – endo effect zone,
3. *T* = 306-562 °C – area of monotonic increase of the heat flux function,
4. *T* = 562-653 °C – exo effect zone,
5. *T* = 653-700 °C – area of monotonic increase of the heat flux function.

When analyzing the thermal regions, it was found that the heat flow function slowly increases in the temperature range *T* = 30-223 °C. At *T* = 223 °C, a sharp decrease in the cost of the heat flow is observed. This process occurs mainly during the absorption of energy. It is known that energy absorption is mainly observed in chemical reactions. This effect is due to the decomposition of $Al(HO)_3$ compounds into water molecules and oxygen atoms, leaving Al nanoparticles. The chemical reaction of the process can be represented as follows:

$$4Al(HO)_3 \rightarrow 4Al + 6H_2O\uparrow + 3O_2\uparrow$$

This effect ended at T = 306 °C. The center of the effect appeared at T = 278 °C, as shown in Figure 2. The sample remaining in the system after this temperature corresponds to pure Al nanoparticles. The phase corresponding to $Al(HO)_3$ compounds completely disappeared.

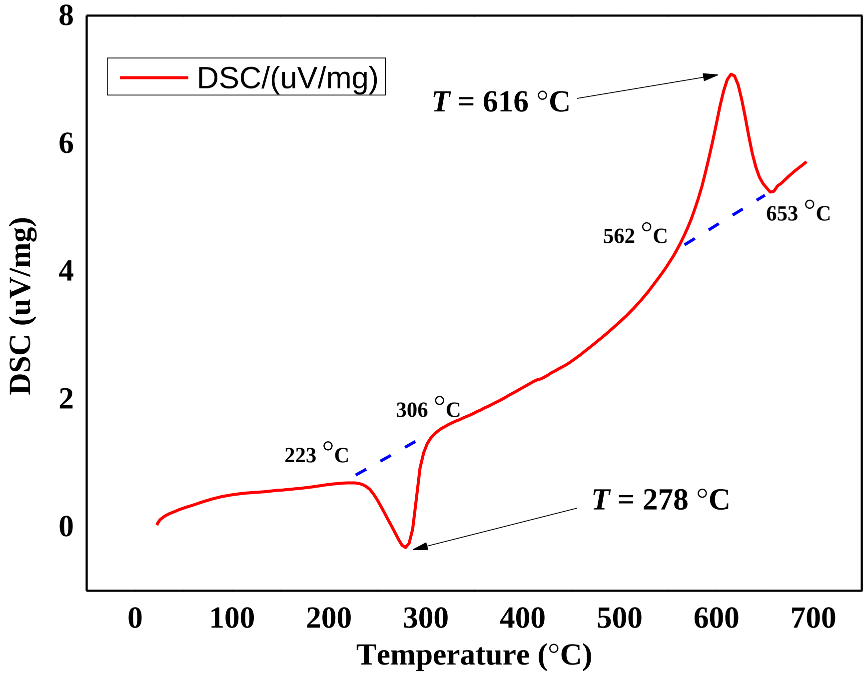

Fig. 2. DSC spectrum of Al nanoparticles in the high-temperature region

After the completion of the endoeffect, in the temperature range T = 223-306 °C, a region of monotonous increase in the heat flux function began. An analysis of the DSC spectrum of Al nanoparticles showed that a new effect is observed at T = 562 °C. There was a jump in the value of the heat flux function, which is consistent with the effects of energy dissipation. This effect corresponded to the exoeffect arising at the central temperature T = 616°C. Such effects arise mainly during phase transitions. It has been established that this effect corresponds to the melting of Al and that a phase transition from solid to liquid occurs. The phase transition ends at T = 653°C. After the phase transition in the region T = 653-700 °C, a monotonic increase in the heat flux function occurred, which corresponds to a change in the heat flux function for Al in the liquid phase.

As a result of DSC analysis, it was found that $Al(HO)_3$ compounds present in Al nanoparticles at room temperature decompose and disappear at high temperatures. In the temperature range T = 306-562 °C, a single-phase system was observed, consisting only of Al nanoparticles. Melting occurred at higher temperatures.

4. Conclusion

The thermophysical properties of Al nanoparticles have been studied. The physicochemical properties of nanoparticles with a size of d = 40-60 nm in the region of high temperatures have been studied. The studies were carried out in the temperature range *T* = 30-700 °C. A Differential Scanning Calorimeter (DSC) was used as a research method. In the DSC spectrum obtained in the high-temperature region, 5 different intervals were observed. It has been found that, under the endoeffect with a central peak *T* = 278 °C, the $Al(HO)_3$ compounds are destroyed and a single-phase system consisting of Al nanoparticles is formed. At *T* = 616 °C, an endoeffect appeared, and this effect corresponded to melting.

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