INVESTIGATION AND CHARACTERIZATION OF METALLIC NANO ALUMINUM POWDER BY ANALYTICAL METHODS

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Abstract. Scanning electron microscope (SEM), X-ray diffraction (XRD) analysis, Raman spectrometry, and Fourier-transform infrared spectroscopy methods are used to study the characterization properties of nano aluminum powder. The determination of nano aluminum powder particle size distribution at the 40-60 nm. The results of the XRD analysis determined the space group of the sample, the parameters of the crystal structure, and the distance between in the atoms. It was determined that the metallic pure nano aluminum sample face-centered cubic crystal structure. The FCC crystal structure of aluminum viewed with [111] pointing up exposes the ABCABC stacking of close-packed atoms. Besides, the results of Raman spectrometry and Fourier-transform infrared spectroscopy determined the vibration frequencies of the atoms, as well as the functional groups. The force between atoms was determined based on the frequencies of motion of the new functional groups.

Keywords: Nanomaterials, analytical methods, XRD-analysis, Raman spectrometry, FTIR.

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1. Introduction

In recent years, micron-sized aluminum powder is one of the most widely used examples in various fields of technology (Yıldırım et al., 2016; Beril Tugrul et al., 2016; Buyuk et al., 2013). The oxidation process in the micro-sized aluminum compounds expands that potential exploitation (Buyuk & Beril Tugrul, 2014; Tashmetov et al., 2019; Buyuk et al., 2014). The solution to current problems with aluminum and the current research results show that the addition of composite materials increases the rate of combustion and specificity. Also used in high concentrations to increase explosive properties and underwater performance (Buyuk et al., 2014). When it comes to crystallography and discussion of aluminum and its alloys by various analytical methods, it appears to have a wide range and a lot of promising materials (Mirzayev, 2020). In the research study, different approaches were chosen for the investigation of nano-aluminum by analytical methods (Mirzayev et al., 2018). This means that the focus is on pure aluminum, regardless of the alloys. The main research discussion on nano-aluminum is based on four topics (Hashimov et al., 2019; Buyuk & Beril Tugrul, 2014). The nature of the crystal structure and effective ways to describe it as a space group, the study of the dynamics of the lattice parameter, Study of the dynamics of particle distribution in nano aluminum compounds using the SEM analysis, Investigation of the amplitude of atomic vibrations in a sample over a wide frequency range using Raman spectroscopic studies and The nature of the functional group's ratios

between aluminum-aluminum atoms has been studied. All of these analysis methods have led to the identification of interatomic relationships and a detailed study of their characteristics. From the crystallographic and optical characteristics of interatomic communications, it can be considered that it is the basis that governs all other aspects of material and structure. Only after considering the four aspects of crystallography of pure aluminum given above, it is expected to change depending on the new external influencing factors.

2. Materials and methods

The aluminum nanopowder with a purity of sample 99.9 %, bulk density of 0.08 g/cm³, specific surface area 20-48 m²/g, and particle size to 40-60 nm (SkySpring Nanomaterials, Inc. USA) was used as a sample (Buyuk & Beril Tugrul, 2014). The structural parameters of samples were measured using the XRD EMPYREAN PANalytical diffractometer in CuK α radiation $\lambda = 1.5406$ Å. In the process of the experiment, in the tube of the diffractometer with copper anode U = 40 kV voltage and I = 40 mA current was generated. The Rietveld analysis of X-ray diffraction patterns has been carried out by the FullProf software package (Mirzayev et al., 2020; Ozyurt et al., 2015). Raman spectroscopy experiments were performed using an INTEGRA Spectra LS PNL instrument at room temperature. The Raman spectrometer SOLAR TII, which was excited with the 633 nm Helium-Neon laser (up to 35 mW output power), the wavelength resolution 0.03 nm, was used in the experiments. The laser beam was focalized onto a spot with a diameter of ~3 µm (Mirzayev et al., 2018). FTIR experiment measurements were performed The Thermo ScientificTM SmartTM iTX ATR Accessory instrument and selected the diamond crystal type for solids state samples (Mirzayev et al., 2019).

3. **Results and discussion**

In the Fig.1 showed the SEM image of the pure metallic nano aluminum compound.

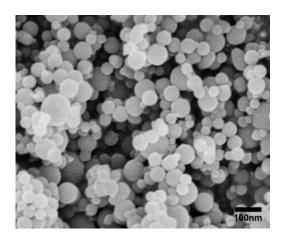


Fig. 1. SEM images of nano aluminum powder sample and particle size distribution at the 40-60 nm

Formation structures of nanomaterials are formed by different mechanisms and depending on different synthesis methods. In the nano-aluminum compound, it was

found that the dynamics of the particle distribution chancing from 40 to 60 nm range, although the dynamics were different. The results of the study are fully consistent with the technical passport provided by the company that performed the synthesis (SkySpring Nanomaterials, Inc. USA) (Mirzayev *et al.*, 2019; Mirzayev, 2020; Zaim, *et al.*, 2016). The mobilization of nanoparticles with a small surface area is also clearly seen in Fig.1. The high value of the specific surface area leads to the ability to react actively. On the other hand, studies have shown that the specific surface area of a nano-aluminum compound with a surface area of 40-60 nm is 30-57 m²/g. The results obtained from the value of the specific surface area are consistent with the literature results (Zaim, *et al.*, 2016; Mirzayev *et al.*, 2020; Beril Tugrul *et al.*, 2015; Demir *et al.*, 2019). In Fig. 2 shown X-ray diffraction spectra of the crystal structure of pure metallic nano aluminum sample.

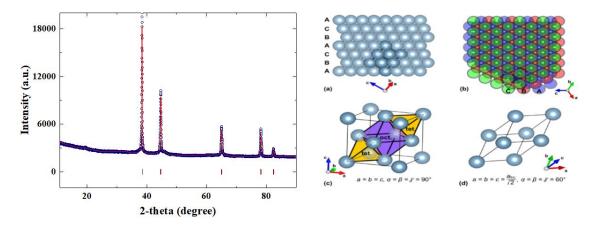


Fig. 2. X-ray diffraction spectra of the nano aluminum sample

Determined the nano aluminum powder in its pure form has a face-centered cubic (FCC) crystal structure. Close-packed arrangement with a layer sequence of ABCABC (Demir *et al.*, 2019) and this is illustrated in Fig. 2. In close-packed structures also both hexagonal close-packed, HCP, and FCC, there are always twice as many tetrahedral interstices as there are octahedral ones. In Fig.3 showed the Raman spectrum of the pure metallic nano aluminum sample.

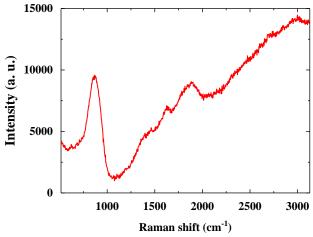


Fig. 3. Raman spectra of the nano aluminum sample

It was determined three major peaks around at 866 cm⁻¹, 1627 cm⁻¹, and 1891 cm⁻¹ in the Raman spectrum. The spectrum shows vibrational modes of Al-Al bonds in the region between 866 cm⁻¹ Raman shifts. In addition, it can be assigned to Al-C interactions 1627 cm⁻¹ disorder aluminum (Demir et al., 2019; Buyuk, 2019; Ozyurt et al., 2018). A relatively weak peak observed at 1891cm⁻¹ is attributed to longitudinal optical (LO) phonons (Buyuk et al., 2012) (also known as A₁ (LO)) of Al-OH. It can be interpreted as an indication of the stress encountered by a certain bond between atoms and the mass increase of lattice atoms because of the capturing of Al and C, O atoms (Durmaz et al., 2014; Buyuk, 2015; Akkas et al., 2015; Demir et al., 2018; Demir et al., 2017). FTIR studies provide detailed information on the state of chemical bonds in materials, bond energy and frequency, optical density, and amorphization mechanism (Demir et al., 2017). The lack of research in the scientific literature on the optical properties of nano-aluminum has aroused great interest in the optical properties of this material. The results of the analysis of the metallic nano Al sample (FTIR) at room temperature show that the field kinetics of the peaks of TOM (transverse optical mode) and LOM (longitudinal optical mode) vary over a wide temperature range. The dynamics of the interaction of the chemical element Al with various chemical elements and functional groups is also shown (Beril Tugrul et al., 2015; Mirzayev et al., 2018). The FTIR spectrum of a metallic nano Al compound is shown in Fig. 4.

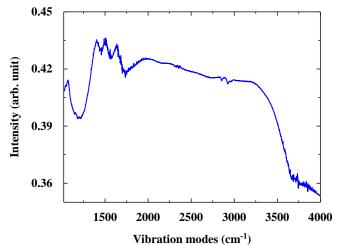


Fig. 4. Fourier-transform infrared spectra of the nano aluminum sample

In the spectrum of the metallic aluminum sample, transverse and longitudinal patterns of 1068 cm⁻¹ Al-H absorption bands are observed at low frequencies. 1402 cm⁻¹ Al-Al transverse optical mode (stretching vibration), 1491 cm⁻¹ Al-C vibration, 1642 cm⁻¹ is accompanied by a change in valence vibration Al-O-Al. In the Al sample, the observation of transverse optical mode and longitudinal optical mode (LOM) is consistent with the references (Demir *et al.*, 2014; Baytaş *et al.*, 2013). Various complex mechanical reactions occur in nano Al surfaces with high surface activity under the influence of environmental factors. Most of the basic reactions of Nano Al compounds in surface chemistry consist of weak interactions with water molecules. Weak absorption spectra in the absorption bands indicate the presence of crystalline water in the structure. A comparative analysis of the oscillations of water in the region of valence and deformation shows that the oscillations lead to transformation. At 2855 and 2931 cm⁻¹, the fashions of the first [OH] groups are noted in very small cases.

Comparative analysis with the results of the literature shows that [Al-OH]⁻ shows chemical bonds. All this is due to the further decomposition of functional [OH]⁻ groups on the surface of nano Al.

4. Conclusion

In this study, aluminum nanopowder with a purity of sample 99.9 %, bulk density of 0.08 g/cm³, specific surface area 20-48 m²/g, and particle size to 40-60 nm was used. It was found that the dynamics of the particle distribution chancing from 40 to 60 nm range, although the dynamics were different. Determined the nano aluminum powder in its pure form has a face-centered cubic (FCC) crystal structure. Raman spectrum of the pure metallic nano aluminum sample was determined three major peaks around at 866 cm⁻¹, 1627 cm⁻¹, and 1891 cm⁻¹, and spectrum shows vibrational modes of Al-Al bonds. FTIR spectrum of a metallic nano Al compound is showed in the aluminum sample, transverse and longitudinal patterns of 1068 cm⁻¹ Al-H absorption bands are observed at low frequencies and 1402 cm⁻¹ Al-Al transverse optical mode (stretching vibration), 1491 cm⁻¹ Al-C vibration, 1642 cm⁻¹ is accompanied by a change in valence vibration Al-O-Al.

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