

SPECTROSCOPIC INVESTIGATION OF THE INFLUENCE OF ALUMINUM ADDITION ON CHARACTERISTIC FEATURES OF ALKALI BOROSILICATE GLASSES

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The influence of aluminum on the structure of alkali borosilicate glasses with various ratios of network former cations and modifier cations has been studied with the use of vibrational spectroscopy. It has been found that addition of modest amounts of aluminum to borosilicate glasses decreases the difference of sodium and potassium distribution between silicon- and boron-containing structural units of the glasses. This fact allows consideration of aluminum as an additive contributory to homogeneity of borosilicate glasses containing both sodium and potassium, as well as increase in thermal and chemical stability of matrix materials based on such glasses.

Keywords: borosilicate glass, spectroscopy, structure, aluminum.

Introduction

During investigation of structural peculiarities of alkali borosilicate glasses with the use of vibrational spectroscopy and NMR spectroscopy the significant difference of sodium and potassium distribution between silicate and borate structural units has been established [1, 2], which determines greater depolymerization of anion structure of sodium-containing borosilicate glasses compared to potassium glasses of similar composition. This explains appearance of structural heterogeneity in the structure of borosilicate glasses containing both sodium and potassium ions [3], besides, it negatively affects physicochemical characteristics of matrix materials based on them [4].

With the aim of looking for additives increasing homogeneity of alkali borosilicate glasses, the influence of aluminum addition on structural features of sodium and potassium borosilicate glasses has been studied by vibrational spectroscopy at various ratios of network former cations and modifier cations. The choice of aluminum as an additive has been caused by the assignment of aluminum to network former cations, highly active in distribution of sodium and potassium ions, which manifest themselves as modifier cations in the glass structure [5–8]. Addition of aluminum to glass composition suggests redistribution of modifier cations between structural units due to their participation in compensation of electrical charge for the AlO_4 tetrahedrons.

Experimental

Aluminum-free and aluminum-containing sodium and potassium borosilicate glasses of the chosen composition were synthesized with the use of following reactants: analytical grade SiO_2 , high purity grade B_2O_3 , chemical purity grade Al_2O_3 , chemical purity grade Na_2CO_3 and K_2CO_3 , according to the procedure described in [2, 9]. Table 1 contains the composition of the synthesized glasses and their reference designations. The investigation of the characteristic structural features was carried out by infrared spectroscopy (IR) and Raman spectroscopy. IR transmission spectra were registered on the single-beam Fourier-transform IR spectrometer Nicolet 6700 Thermo Scientific with the use of KBr pellet pressing technique. The iHR 320 Labram spectrometer with Olympus BX41 microscope was used for registration of Raman spectra. The obtained spectra are shown in Fig. 1–4.

Table 1

Chemical composition of the synthesized glasses

Sample	Composition, mole fractions
15N35B	$0.15\text{Na}_2\text{O} \cdot 0.35\text{B}_2\text{O}_3 \cdot 0.5\text{SiO}_2$
35N15B	$0.35\text{Na}_2\text{O} \cdot 0.15\text{B}_2\text{O}_3 \cdot 0.5\text{SiO}_2$
15K35B	$0.15\text{K}_2\text{O} \cdot 0.35\text{B}_2\text{O}_3 \cdot 0.5\text{SiO}_2$
35K15B	$0.35\text{K}_2\text{O} \cdot 0.15\text{B}_2\text{O}_3 \cdot 0.5\text{SiO}_2$
A15N35B	$0.9(0.15\text{Na}_2\text{O} \cdot 0.35\text{B}_2\text{O}_3 \cdot 0.5\text{SiO}_2) + 0.1\text{Al}_2\text{O}_3$
A35N15B	$0.9(0.35\text{Na}_2\text{O} \cdot 0.15\text{B}_2\text{O}_3 \cdot 0.5\text{SiO}_2) + 0.1\text{Al}_2\text{O}_3$
A15K35B	$0.9(0.15\text{K}_2\text{O} \cdot 0.35\text{B}_2\text{O}_3 \cdot 0.5\text{SiO}_2) + 0.1\text{Al}_2\text{O}_3$
A35K15B	$0.9(0.35\text{K}_2\text{O} \cdot 0.15\text{B}_2\text{O}_3 \cdot 0.5\text{SiO}_2) + 0.1\text{Al}_2\text{O}_3$

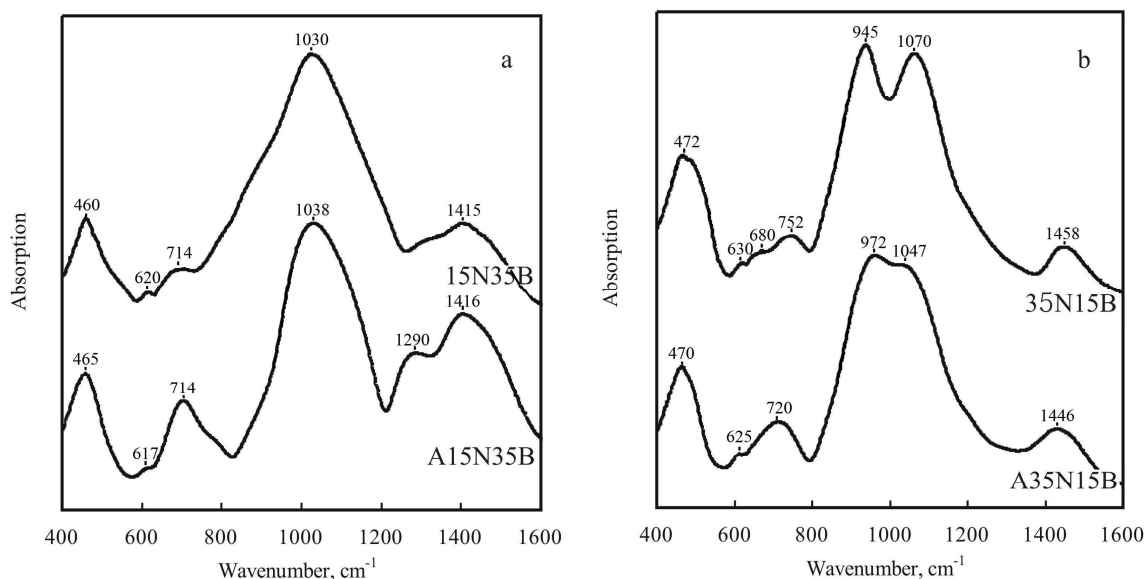


Fig. 1. IR spectra of the sodium glasses:
 a – glasses with low content of sodium and high content of boron, b – glasses with high content of sodium and low content of boron (designations of the samples correspond to Table 1)

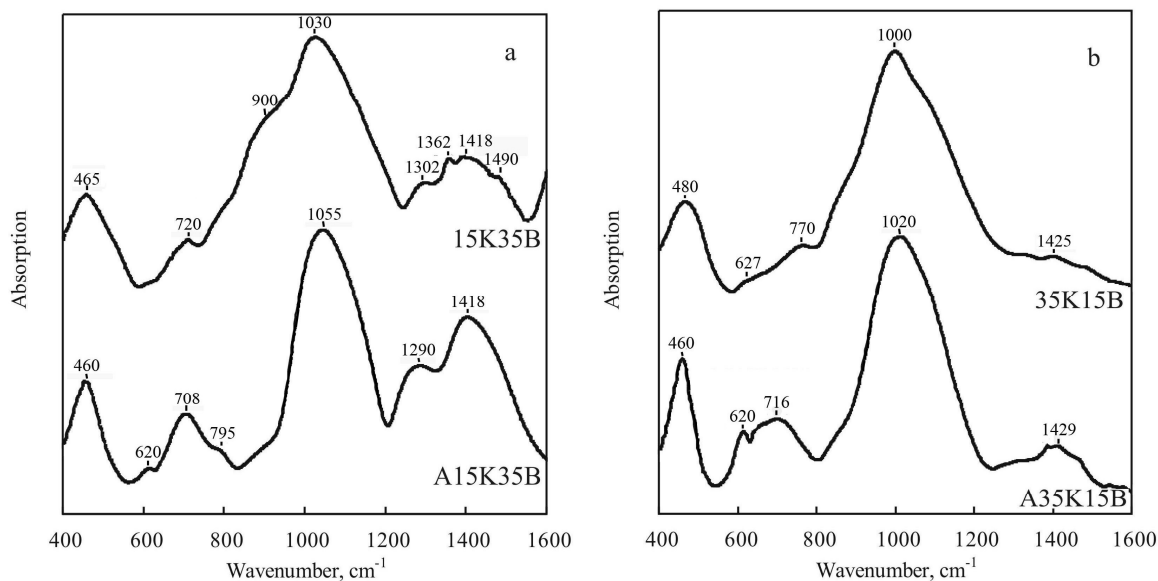


Fig. 2. IR spectra of the potassium glasses:
 a – glasses with low content of potassium and high content of boron, b – glasses with high content of potassium and low content of boron (designations of the samples correspond to Table 1)

Results and Discussion

All IR spectra of the synthesized glasses (Fig. 1 and 2) contain bands with maxima near 460–480, 610–800, 800–1200 and 1350–1490 cm^{-1} . In the IR spectra of aluminum-free sodium glasses (Fig. 1) the increase of alkali metal content and the decrease of boron content leads to the increase of band intensity for the maximum near 460–480 cm^{-1} , the change of band shape for the maximum near 610–800 cm^{-1} , and the decrease of band intensity for the maximum near 1350–1490 cm^{-1} . In the spectrum of 35N15B glass the following features are observed: the band with the maximum near 1030 cm^{-1} , which dominates in the spectrum of 35N15B glass, separates into two components with maxima near 945 cm^{-1} and 1070 cm^{-1} .

Changes in the IR spectra of aluminum-free potassium glasses with similar changes in composition are also related to the change in band shape in the range 800–1200 cm^{-1} and the decrease of band intensity for the maximum near 1350–1490 cm^{-1} (Fig. 2). At low content of potassium and high content of boron in the glass composition (15K35B) the band with maximum near 1030 cm^{-1} and the plateau near 900 cm^{-1} are observed in the range 800–1200 cm^{-1} . At the increase of potassium content and the decrease of boron content in the glass composition (35K15B) the maximum of this band shifts to 1000 cm^{-1} with significant change in the position and intensity of its branches.

The IR spectra of all synthesized aluminum-containing sodium and potassium glasses (A15N35B, A35N15B, A15K35B, A35K15B) are characterized by higher intensity of bands with the maxima near 610–800 cm^{-1} , significant changes in band shape in the range 800–1200 cm^{-1} , and the increase of band intensity for the maximum near 1350–1490 cm^{-1} . It is especially noticeable in the spectra of glasses with low content of alkali metal and high content of boron (Fig. 1 and 2).

IR absorption near 460–480 and 1050–1090 cm^{-1} is related to deformation and asymmetric stretching vibrations of Si–O–Si(Al) bonds in complex silicate anions [10–12], its intensity in spectra is determined by the content of SiO_2 in the glass composition. The band near 610–800 cm^{-1} makes itself evident as the superimposed on one another bands due to symmetric vibrations of Si(Al)–O–Si bonds, asymmetric stretching vibrations of B–O–B bonds, deformation vibrations of B–O bond in the BO_3 triangles, and vibrations of the bonds in the AlO_4 tetrahedrons. This complicates the use of absorption in this range for discussion of changes in the glass structure. Absorption in the range 940–980 cm^{-1} is related to deformation and asymmetric stretching vibrations of B–O bond in the BO_4 tetrahedra, which allows estimating the change of boron ions content in the tetrahedral coordination [10, 12]. The band in the range 1350–1490 cm^{-1} is related to deformation and stretching vibrations of B–O bonds of the BO_3 triangles. Decrease of its intensity reflects the decrease of the tri-coordinated boron content in the glass structure [10, 11]. Changes in IR spectra of glasses observed at aluminum addition correlate with the increase of the fraction of BO_3 triangles, the decrease of BO_4 tetrahedra, and the changes in the nearest environment of silicon atoms through the development of AlO_4 tetrahedra in the glass structure, as well as the formation of bridging intertetrahedral Si–O–Al bonds.

The Raman spectra of all synthesized aluminum-free sodium and potassium glasses strongly differ from each other. In the Raman spectrum of 15N35B glass (Fig. 3a) there are intensive bands with maxima at 510 and 1150 cm^{-1} , as well as the set of closely spaced bands of lower intensity with the maxima near 630, 700, 760 and 800 cm^{-1} . In the Raman spectrum of 35N15B glass (Fig. 3b) intensive bands with the maxima near 600, 955 and 1090 cm^{-1} are observed. In the Raman spectrum of 15K35B glass (Fig. 4a) we have registered the intensive band with the maximum at 510 cm^{-1} , the set of closely spaced bands of lower intensity with the maxima near 630, 700, 770 and 800 cm^{-1} , as well as three weak broad bands with the maxima near 940, 1150 and 1450 cm^{-1} . The Raman spectrum of 35K15B glass (Fig. 4b) is characterized by the band with the maximum near 600 cm^{-1} and the plateau near 530 cm^{-1} , as well as the narrower intensive band 1095 cm^{-1} and weak bands with the maxima near 940 and 1450 cm^{-1} .

Addition of aluminum to glass composition significantly influences the position and intensity of the Raman bands. In the Raman spectrum of aluminum-containing sodium glass A15N35B (Fig. 3a) the significant decrease of the band intensity with the maxima near 630, 700 and 1150 cm^{-1} is observed. In spite of obvious differences in the spectra of parent glasses 15N35B and 15K35B, taking into account slight differences in intensity ratios and extra plateaus, the spectrum of aluminum-containing potassium glass A15K35B (Fig. 4a), has the similar appearance to the Raman spectrum of A15N35B glass. In the low-frequency (300–800 cm^{-1}) part of the Raman spectrum of aluminum-containing sodium glass A35N15B (Fig. 3b) the band of complex shape with the maximum near 510 cm^{-1} and plateaus near 570,

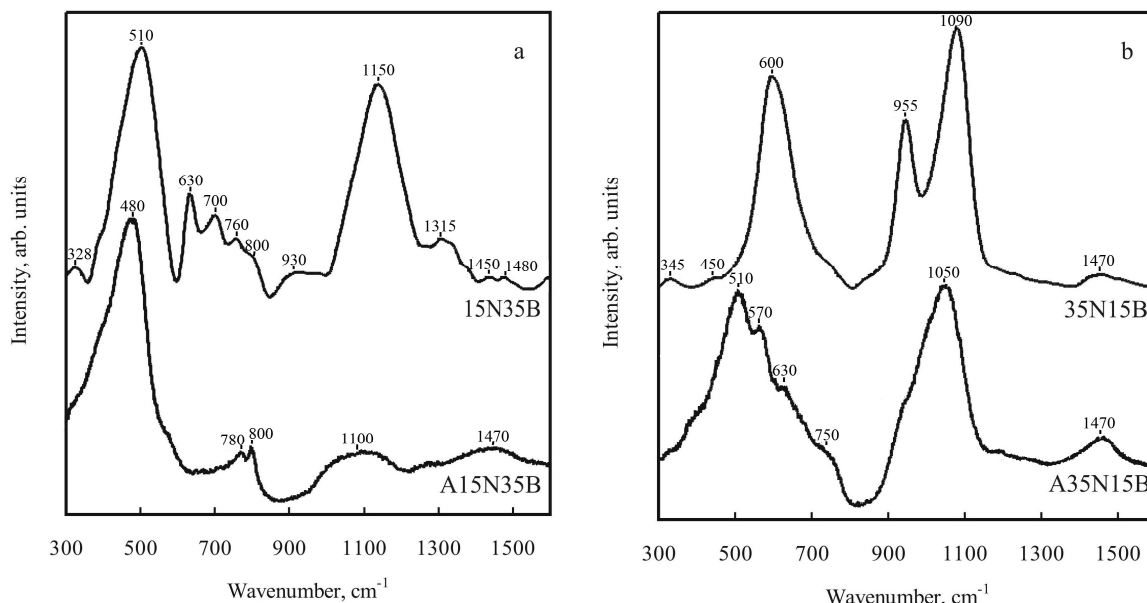


Fig. 3. Raman spectra of the sodium glasses:
a – glasses with low content of sodium and high content of boron, b – glasses with high content of sodium and low content of boron (designations of the samples correspond to Table 1)

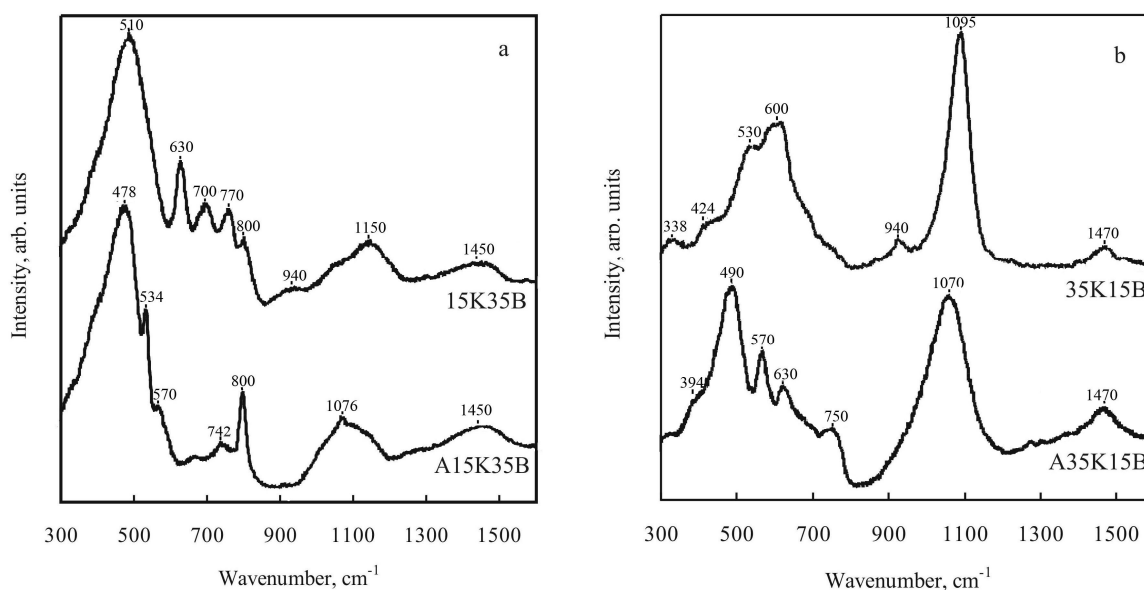


Fig. 4. Raman spectra of the potassium glasses:
a – glasses with low content of potassium and high content of boron, b – glasses with high content of potassium and low content of boron (designations of the samples correspond to Table 1)

630 and 750 cm^{-1} is registered. In the high-frequency part of the Raman spectrum of this glass the band with the maximum near 1050 cm^{-1} is observed, with strongly expressed asymmetry as viewed from low wavenumbers. Besides, the band of low intensity with the maximum 1470 cm^{-1} is clearly defined in the spectrum. The Raman spectrum of aluminum-containing potassium glass A35K15B (Fig. 4b) is distinguished from the spectrum of A35N15B glass only by the slight shift of the band maxima (510 \rightarrow 490 cm^{-1} and 1050 \rightarrow 1070 cm^{-1}) and better defined plateaus near 570, 630 and 750 cm^{-1} . Hence the aluminum addition increases resemblance of the Raman spectra of sodium and potassium glasses of similar composition, both with low and high content of alkali metal.

The bands with the maxima near 400–600 cm^{-1} in the low-frequency part of the Raman spectra of borosilicate glasses are related to symmetrical stretching and deformation vibrations of Si–O–Si(Al) and Si(Al)–O–B bridging bonds in anions with complex structure [13, 14]. High intensity of these bands,

observed in the Raman spectra of all investigated glasses indicate the relatively high polymerization degree of their anion structure. The band with the maximum near 630 cm^{-1} reflects the vibrations of mixed silicon-boron rings, and decrease of intensity for this band indicates decrease of boron content in the glass composition or at decrease of its participation in formation of the mixed borosilicate rings. The bands in the range $700\text{--}800\text{ cm}^{-1}$ are related to vibrations of B–O bonds included in the BO_4 tetrahedra, which helps us to use these bands for estimation of the boron state in the glass structure. The bands in the range $950\text{--}1150\text{ cm}^{-1}$ are related to the manifestation of stretching vibrations of the Si–O– nonbridging bonds in Q^2 and Q^3 structural units [15]. Their presence in the glass spectra indicates participation of sodium and potassium ions in coordination of the nonbridging bonds, while the observed change of symmetry in these bands at aluminum addition arises from the presence of the AlO_4 tetrahedrons in the nearest environment of those structural units. The band with the maximum near 1470 cm^{-1} reflects the vibrations of the BO_3 structural units. Low intensity of this band in the spectra of borosilicate glasses generally points at boron in tetrahedral coordination (BO_4) dominating in the structure of such glasses.

Conclusion

The characteristic features of obtained Raman spectra indicate the difference in distribution of modifier cations between various kinds of structural units in the structure of aluminum-free and aluminum-containing sodium and potassium borosilicate glasses. In aluminum-containing glasses the development of the AlO_4 structural units, participating in formation of the bridging Si–O–Al bonds, is observed in the studied composition range. The fraction of the BO_4 tetrahedra decreases, while the fraction of the BO_3 triangles, which form the isolated boron ring structures, increases. Mostly these changes appear in the glasses with low content of alkali metals and high content of boron. In the glasses with high content of alkali metals and low content of boron the changes touch upon the silicate part of the glass structure, too. The decrease of the fraction of silicate structural units with nonbridging oxygen atoms (Q^n) is under way, as well as the decrease of the fraction of nonbridging bonds in the composition of the borate structural units. It correlates with the decrease of participation of alkali metal cations in the coordination of the charge of boron-containing structural units BO_4 , which decreases the difference in the distribution of sodium and potassium between silicate and borate structural units and enhances the structural semblance of sodium and potassium glasses with the similar composition. The significant fraction of sodium and potassium ions in the structure of aluminum-containing glasses participate in the coordination of the charge of aluminum in tetrahedral coordination, which corresponds to stronger binding of Na^+ and K^+ and promotes increase of thermal and chemical stability of matrix materials based on such glasses.

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СПЕКТРОСКОПИЧЕСКОЕ ИССЛЕДОВАНИЕ ВЛИЯНИЯ ДОБАВОК АЛЮМИНИЯ НА СТРУКТУРНЫЕ ОСОБЕННОСТИ ЩЕЛОЧНЫХ БОРОСИЛИКАТНЫХ СТЕКОЛ

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Методами колебательной спектроскопии изучено влияние алюминия на структуру щелочных боросиликатных стекол с разным соотношением катионосеткообразователей и катионов-модификаторов. Установлено, что при добавлении небольшого количества алюминия в состав стекол происходит уменьшение различия в распределении натрия и калия между боратными и силикатными составляющими структуры боросиликатных стекол. Это позволяет рассматривать алюминий в качестве добавки, способствующей увеличению однородности боросиликатных стекол, одновременно содержащих натрий и калий, и повышению термической и химической устойчивости матричных материалов, создаваемых на основе этих стекол.

Keywords: боросиликатные стекла, спектроскопия, структура, алюминий

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