Infrared Spectroscopic Investigation of Chemisorption on Chemically Synthesized Aluminum Nitride Nanopowders with 2-Pyrrolidinones

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The chemical nature of a nanoparticle surface is of crucial importance in controlling the interparticle forces during powder processing. As a continuation of a FT-IR spectroscopy study on the surface chemical structure of the chemically synthesized aluminum nitride (AlN) nanopowders, the adsorption of two representative cyclic amides, i.e., *N*-methyl-2-pyrrolidinone (NMP) and 2-pyrrolidinone (NHP), have been investigated. Upon adsorption of these cyclic amides, strong hydrogen bonding between the amide carbonyl oxygen and the surface hydroxyl groups was evident. The unusual thermal stability for the hydrogen bonding observed with NMP was explained as being due to the enhancement by secondary interactions between the NMP amide carbonyl group and the surface Lewis acidic sites, such as Al^{3+}. With the adsorption of NHP at room temperature, besides the formation of strong hydrogen bonding, a striking 100 cm^{-1} shift to higher wavenumbers was observed for both the carbonyl and amide C−N stretching bands. This has been interpreted as the formation of complex coordination between the amide nitrogen and the surface acidic sites. The amide N-coordinated complex plus the carbonyl O-coordinated hydrogen bonding promote a synergistic effect for strengthening the NHP adsorption on the AlN surface.

Introduction

Aluminum nitride (AlN) is of great interest for a wide range of electronic and structural applications.[1−3] The more recent development of nanostructured AlN has the potential of unique properties in comparison with those of the conventional micron-sized materials.[4−6] With the advent of these new types of materials, powder processing has attained increasing interest.[7] The chemical nature of ceramic powder surfaces plays a key role in controlling the interparticle forces. The adsorption of dispersants, surface charge properties, and the suspension stability are often related to the surface chemical properties of the ceramic powders. In the production of advanced and high-performance ceramic materials, especially nanopowders, the characterization and control of the surface chemical properties are therefore of major importance.

We previously reported a comprehensive surface characterization of a chemically synthesized nanostructured AlN powder.[8] It revealed a surface chemical composition that mainly consisted of −OH and $-NH_2$ groups, as well as physisorbed H_2O, CO_2, and NH_3 molecules. Acidic and weak basic surface sites were also evident. In yet another study of a homogeneous dispersion of AlN/polyimide nanocomposite, a dynamically stable suspension has been achieved by using a non-aqueous media.[9,10] The current investigation was aimed at developing a mechanistic interpretation of the observed properties of the AlN suspensions. In particular, the surface chemical interactions of the AlN nanoparticles with *N*-methylpyrrolidinone (NMP) and 2-pyrrolidinone (NHP) were studied by in situ FT-IR analysis. As we demonstrated in our previous work,[8] surface FT-IR analysis is useful in providing information about different types of surface functional groups and the adsorption mechanisms. The IR absorption frequencies of both the surface group and the interacting molecular group are usually shifted when a sorbate molecule interacts with a surface functional group. It is particularly sensitive for high surface area systems, such as

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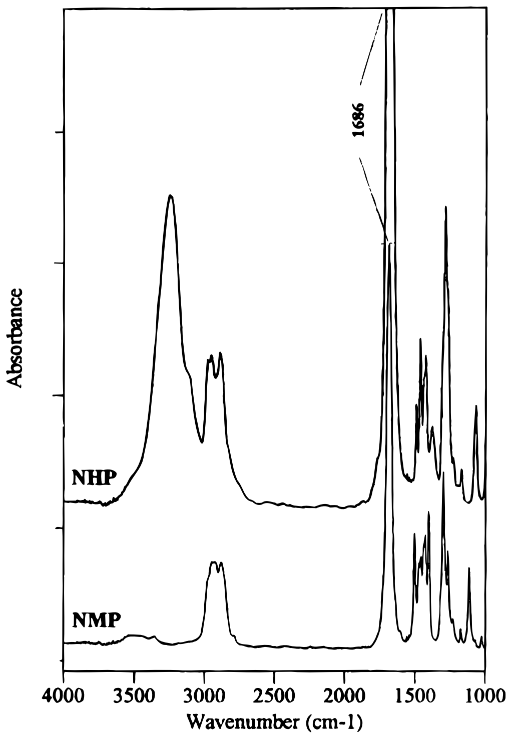


Figure 1. FT-IR spectra of 2-pyrrolidinon (NHP) and *N*-methyl-2-pyrrolidinone (NMP).

nanopowders. In addition, temperature effects can also be investigated via this technique.

Experimental Section

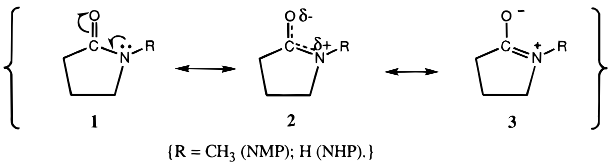
Nanostructured AlN powder sample was synthesized and stored in a protective environment as described previously.[8] The morphology of the nanopowder has been thoroughly characterized.[8] The surface area of the AlN powder was determined as 43 m^2/g by BET measurement (Omnisorb cx100) by nitrogen adsorption. A 200 mg of the AlN sample was outgassed at 300 °C for 24 h prior to performing the BET analysis. The average surface area was based on the linear portion of the BET plot with P/P_0 range from 0.006 to 0.3.

N-Methyl-2-pyrrolidinone (Aldrich, 99%) and 2-pyrrolidinone (Aldrich, 99+%) were used without further purification. The FT-IR spectra of each compound has been examined to match the vibrational assignments in the literature.[11] For comparison with the adsorbed species, the FT-IR spectra of liquid NMP and NHP are shown in Figure 1.

All the surface FT-IR experiments were recorded with a Nicolet 5DX Fourier transform infrared spectrometer that operates from 4000 to 400 cm^{-1} with a 4 cm^{-1} resolution. A thin disk of the sample was prepared by pressing the pure powder onto a stainless steel grid. Only a low pressure was applied in order to keep the AlN grains loose enough to allow the gaseous adsorbate to have a maximum contact surface. The sample disk was then placed inside a custom designed cell[8] which allowed heating under a vacuum, controlled pressure of gases or vapors of various species including reactive probe molecules. The cell was centered in the sample compartment of the spectrometer, so as to enable the in situ recording of spectra at a temperature range from ambient to 873 K.

The sample pellet was activated under vacuum (5×10^{-5} mbar) at 873 K for 1 h before the introduction of probe molecules. The activation step resulted in the desorption of physisorbed or weakly chemisorbed species. Hence the activated surface was no longer in an equilibrium state. When probe molecules were in contact with the surface, they would adsorb on the activated sites. To eliminate a possible temperature effect, all the spectra were recorded at room temperature.

Scheme 1



Results and Discussion

The cyclic structure of pyrrolidinones results in the amide group being less screened by neighboring atoms and groups compared to other amides. Hence they undergo strong specific interactions with different species and form complexes.[12] These cyclic amides have been intensively studied principally with the polymerization of lactams.[13] In general, pyrrolidinones (**1**) have amide groups with a partial double bond character for the C−N bond, resulting from the delocalization of the nitrogen lone-pair electrons (p-electrons) into the π-system of the carbonyl double bond as shown in Scheme 1. They give rise to a planar structure (**2**) for the atoms connected to C and N.[13] As a result, the C−N and C−O bonds of pyrrolidinones have bond lengths intermediate between the two contributing resonance (mesomeric) forms (**1** and **3**). They are therefore usually weakly amphiprotic and can form hydrogen-bonded structures or coordinating complexes. The carbonyl oxygen atom of pyrrolidinones is the acceptor site for hydrogen bonding with many protic and aprotic donor molecules. In complex compounds with transition or non-transition metals the carbonyl oxygen coordination is favored,[14] while pyrrolidinone salts by nitrogen do form, as reported in the study of reactions of lactams with alkaline metals.[13]

The current investigation is intended to impart the details of surface interactions of the AlN nanoparticles during the adsorption of these cyclic amides. The assignments of the vibration frequencies for the as-synthesized AlN and the sample activated at 873 K were reported previously.[8] The prominent surface species are summarized as follows: The complex band in the highest wavenumber region (~3740 cm^{-1}) was assigned to the $\nu(OH)$ stretching vibrations of several types of Al−OH hydroxyl groups. The broad band at ~3200 cm^{-1} corresponded to the $\nu(NH)$ stretching vibrations of NH_2 groups, which is also associated with the $\delta(NH_2)$ band at 1543 cm^{-1}. Several bands in the 1200−1600 cm^{-1} region are due to overtones or combinations of vibration modes of the bulk AlN. The difference spectrum of the activation step depicts the desorption of the physisorbed species. By analyses of the corresponding negative bands,[8] the species that can be detected include water from the ambient atmosphere, ammonia originating from the synthesis process, and atmospheric

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Table 1. Assignments of the Vibrational Frequencies of Adsorption of NMP on the AlN Surface[a]

T (K)	freq (cm^{-1})	intensity	assignment
room temperature	~3740 (difference spectrum)	negative band	losing "free" stretching OH band on the surface
	3600−3000	strong, broad	hydrogen-bonded OH stretching on the surface
	2958, 2935, 2883	s	$\nu_{asym}(CH_3)$; $\nu_{sym}(CH_3)$; $\nu_{sym}(CH_2)$
	1665	vs	$\nu(C{=}O)$
	1511	s	$\nu(C'{-}N)$; $\delta_{sym}(CH_3)$; CH_2 scissor
	1478	m	$\delta_{asym}(CH_3)$
	1467, 1446, 1428	m	CH_2 scissor
	1408	s	CH_3 sym and asym deformation; CH_2 wag, $\nu(C{-}N)$; $\nu(N{-}CH_3)$
	1305, 1265, 1231, 1177	s, m	CH_2 wag and twist
	1117	m	$\nu(C{-}N)$; $\nu(N{-}CH_3)$; CH_3 sym rock
423	~3740 (difference spectrum)	positive band	retrieve "free" stretching OH band on the surface
	2968, 2943, 2893	m	$\nu_{asym}(CH_3)$; $\nu_{sym}(CH_3)$; $\nu_{sym}(CH_2)$
	1658	vs	$\nu(C{=}O)$
	1519	s	$\nu(C'{-}N)$; $\delta_{sym}(CH_3)$; CH_2 scissor
	1484	w	$\delta_{asym}(CH_3)$
	1459	w	CH_2
	1416	m	CH_3 sym and asym deformation; CH_2 wag, $\nu(C{-}N)$; $\nu(N{-}CH_3)$
	1328, 1315, 1261	w	CH_2
	1117	m	$\nu(C{-}N)$; $\nu(N{-}CH_3)$; CH_3 sym rock
573	2966, 2948, 2895	m	$\nu_{asym}(CH_3)$; $\nu_{sym}(CH_3)$; $\nu_{sym}(CH_2)$
	1658	vs	$\nu(C{=}O)$
	1517	s	$\nu(C'{-}N)$; $\delta_{sym}(CH_3)$; CH_2 scissor
	1484	w	$\delta_{asym}(CH_3)$
	1459	w	CH_2
	1417	m	CH_3 sym and asym deformation; CH_2 wag, $\nu(C{-}N)$; $\nu(N{-}CH_3)$
	1329, 1315, 1262	w	CH_2
	1117	w	$\nu(C{-}N)$; $\nu(N{-}CH_3)$; CH_3 sym rock

[a] Summarized from ref 11. s, m, w = strong, medium, weak; ν = stretch, δ = bending vibrations; C′ = carbonyl carbon.

carbon dioxide. The acidity of the AlN surface was proven by using pyridine as a probe molecule, which showed the presence of two types of Al^{3+} Lewis sites. On the other hand, a specific dissociation mechanism revealed by methanol adsorption was explained as the presence of weakly basic Al_3N sites.[8]

Adsorption of *N*-Methyl-2-pyrrolidinone. NMP behaves as a rigid, nonassociating polar species.[15] It has a more stable amide C−N bond compared to an unsubstituted pyrrolidinone and will not in general polymerize.[13] This is due to the electron-donating effect of the N-substituted methyl group, which increases the resonance stabilization of the amide group. This electron-donating effect also strengthens the basicity of the amide carbonyl group, which promotes a stronger tendency to form a hydrogen bond or coordinate to other Lewis acidic sites.[14]

Figure 2 shows the adsorption spectra of NMP on the activated AlN surface at room temperature and increasing temperature under continuous evacuation. All these spectra are actually depicted by subtracting the spectrum of the activated AlN sample, so that all the vibration bands are attributed only to the surface-adsorbed species and/or to the possibly modified surface groups. The observed vibration frequencies of NMP adsorbed on the AlN-activated surface at room temperature and stepwise elevated temperatures are also summarized in Table 1.

The adsorption of NMP on the nanostructured AlN surface appeared to form a strong hydrogen bond with the surface hydroxyl groups as shown in Figure 2a. Intermolecular hydrogen bonding caused a broad band in the 3000−3500 cm^{-1} region, due to the ν(OH) stretching vibration of the perturbed OH groups,[16] while the negative band at ~3740 cm^{-1} confirmed the decrease or the perturbation of free OH groups.

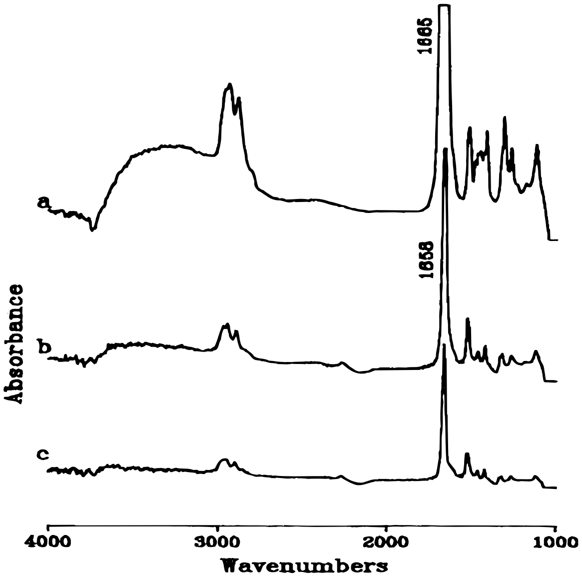

Figure 2. FTIR spectra for the addition of NMP: (a) addition at room temperature and after desorption at (b) 423 and (c) 573 K. All the spectra are subtracted from the surface activation spectrum.

Associated with the surface hydrogen bonding, a shift to lower frequency of the carbonyl band from 1686 cm^{-1} (unperturbed free NMP in liquid phase) to 1665 cm^{-1} was observed, which suggested that the carbonyl oxygen atom was the acceptor site for the hydrogen bonding with the surface OH groups. Considering the overall vibrational bands of the NMP adsorption spectrum (Figure 2a), no other apparent shift was observed to such a great extent from its free-form spectrum.[11] This suggested that the hydrogen bonding was the main interaction occurring during the NMP adsorption onto the AlN surface at room temperature.

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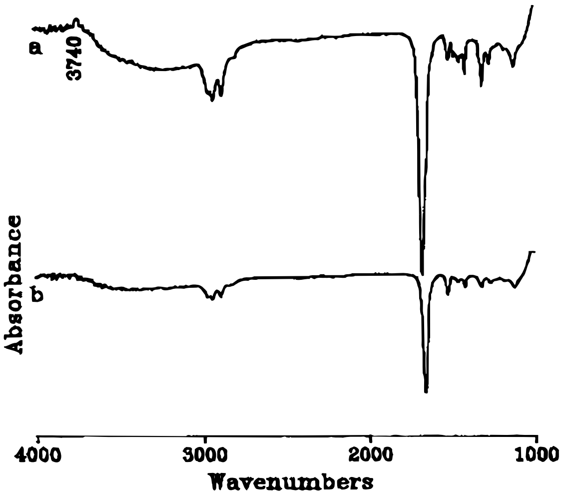
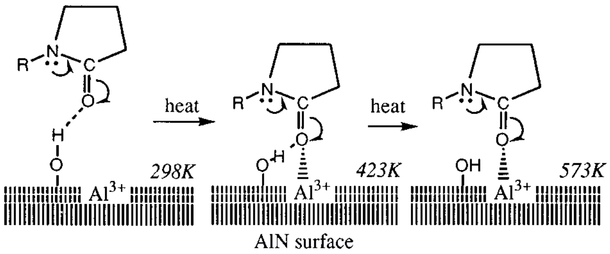


Figure 3. Difference spectra between two desorption steps of NMP (to be compared with Figure 2): (a) from room temperature to 423 K; (b) from 423 to 573 K.

Upon heating to 423 K, the broad hydrogen-bonding band decreased to a great extent (Figure 2b). The difference spectra between the two steps (Figure 3a) also proved this desorption process. Meanwhile, the appearance of a positive band at 3740 cm^{-1} corresponds to the recovery of free surface OH groups. These data showed that the majority of the surface hydrogen bonds were dissociated at this condition. As the temperature was further increased to 573 K, the hydrogen bond was completely dissociated (Figures 2c and 3b). At 423 K, however, the broad peak in 3000−3500 cm^{-1} region was still evident and remained approximately one-fourth of its intensity compared to that in the room-temperature spectrum (Figure 2a,b). Moreover, all of the bands corresponding to the NMP molecule were evident, while only the intensity ratio varied. This indicated an uncommon strength of the surface hydrogen bond, because it is usually completely removed at 423 K in the previously reported methanol adsorption experiment.[8] A careful observation of the carbonyl band revealed a further shift to 1658 cm^{-1} (Figure 2b), which is even lower than that at room temperature (1665 cm^{-1}, Figure 2a). In general stronger intermolecular interactions with carbonyl oxygen will shift the carbonyl band to the lower frequency, yet the exact bonding structure of this type could be more complicated. A rationale to this phenomenon could be that a secondary interaction between the adsorbed NMP molecules and the AlN surface as observed for methanol adsorption on alumina.[17] This secondary interaction could occur because of the increasing temperature, which not only caused the breaking of normal hydrogen bonds but also modified the adsorbed NMP molecules on the surface acidic sites as observed in the case of pyridine adsorption.[8] The formation of such a coordination structure between the surface Lewis sites and NMP molecules could be favorable considering the high electron density of the NMP carbonyl group. Numerous studies on organometallic−NMP complexes also support this hypothesis.[13] Thus, a portion of the hydrogen-bonded carbonyl oxygen of the remaining NMP may be further associated with the surface acidic Al^{3+} sites. These NMP molecules on the surface therefore achieved a stronger association force, that also contributed to the above mentioned carbonyl shift. The proposed mechanism for the secondary interactions is described in Scheme 2.

Scheme 2



The hydrogen bonding is completely dissociated at 573 K (see Figure 2c), and more NMP molecules have been removed after continuous evacuation (Figure 3b), but NMP is still detectable on the surface. Most of the bands corresponding to the NMP molecule still persist, albeit their peak intensities decreased dramatically and the precise fingerprint structure below 1600 cm^{-1} changed only slightly. Because the carbonyl band (1658 cm^{-1}) at this temperature has not changed compared to that of 423 K (see Table 1), it appears that the secondary interaction mentioned above still existed and became the main type of association between the surface and the remaining adsorbed molecules.

Adsorption of 2-Pyrrolidinone. NHP is a useful lactam that has a wide range of applications. Its acidic character, or the tendency to lose a proton, is increased compared to amines, and the basic character weakened.[17,18] The N-coordination to metal cations has been observed and utilized for the study of anionic polymerization of lactams.[17] The byproducts of the coordination reaction were observed as proton acidic character.[17]

Table 2 summarizes the observed vibrational frequencies of NHP adsorbed on the AlN surface at room temperature and 423 K under continuous evacuation. The data were obtained from the difference spectra between the adsorbed and activated sample surface.

The adsorption spectra of NHP at various temperatures, obtained by subtracting a background spectrum of the activated surface, are shown in Figure 4. The formation of a hydrogen bond with the surface OH groups at room temperature is indicated as a broad band in the range 3000−3500 cm^{-1} and a weak negative band around 3740 cm^{-1} (Figure 4a). A shift for the carbonyl band to a lower frequency was observed from 1686 cm^{-1} (for the free form of NHP[15]) to 1667 cm^{-1} (Figure 4a), which is also associated with the formation of hydrogen bonding. The relative weakness of the negative 3740 cm^{-1} band, however, is an indication of less “free” OH groups in association with NHP. Considering the weakened amide carbonyl basicity of NHP as compared to that of NMP, this decreased tendency to form hydrogen bonds is reasonable.

The striking difference between the adsorption of NMP and NHP is that, upon the adsorption of NHP at room temperature, the spectrum showed a group of interesting new bands (see Figure 4a) compared to the spectrum of pure NHP. The most remarkable new band appeared at 1762 cm^{-1}, which can be assigned to the carbonyl C=O stretching vibration. Compared to the

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Table 2. Assignments of the Vibrational Frequencies of Adsorption of NHP on the AlN Surface[a]

T (K)	freq (cm^{-1})	intensity	assignment
room temperature	3700−2800	broad mixed bands	hydrogen-bonded OH on the surface; ν(N−H), ν(C−H)
	1762	s	ν(C=O)
	1667	s	ν(C=O)
	1596	s	ν(C′−N); ν(C−C)
	1463, 1422	m	CH_2 scissor
	1314	s	CH_2 wag
	1187	m	CH_2 twist; ring angle bend
423	2850−2950	m	ν(C−H)
	1654	w	ν(C=O); ν(C′−N); ν(C−C)
	1587	s	$\delta_{asym}(CH_3)$
	1470, 1429	m	CH_2 scissor
	1310	s	CH_2 wag
	1187	w	CH_2 twist; ring angle bend

[a] Summarized from ref 11. s, m, w = strong, medium, weak; ν = stretch, δ = bending vibrations; C′ = carbonyl carbon.

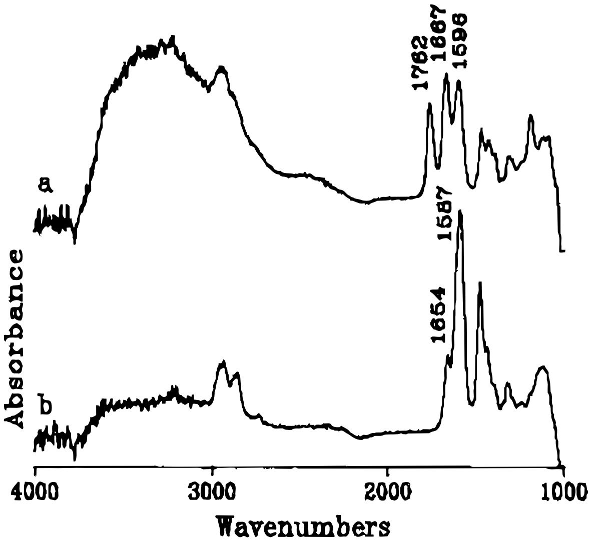
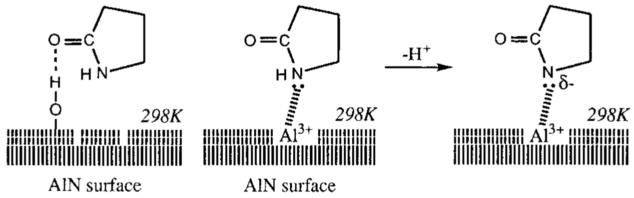


Figure 4. FTIR spectra for the addition of NHP: (a) addition at room temperature; (b) after desorption at 423 K. All the spectra are subtracted from the surface activation spectrum.

Scheme 3



pure NHP spectrum, a total 100 cm^{-1} shift toward the higher wavenumber region was attained. The increase of the bond energy is associated with the strengthening of the double bond feature of the amide carbonyl group. It can be achieved by the complex coordination of the amide N atom adjacent to the carbonyl bond. The nitrogen coordination was verified, since another frequency shift of the characteristic amide band from 1492 cm^{-1} (free form of NHP) to 1596 cm^{-1}, corresponds to the strengthening of C′−N (carbonyl carbon−nitrogen) stretching vibration. As the fundamental discussion made earlier on lactams, this coordination should favor surface acidic sites such as Al^{3+}, as shown in Scheme 3.

The association involved in proton transfer is significant for the processing of ceramic suspensions. Fowkes and co-workers[19] have described that if the concentration of dispersant dissolved in the organic phase is sufficiently high, then dynamic adsorption and desorption occur, and some proton-carrying dispersants will desorb into solution and provide the counterions for the negative charges remaining on the surface. Thus, this will definitely influence the deagglomeration and stabilization mechanism.

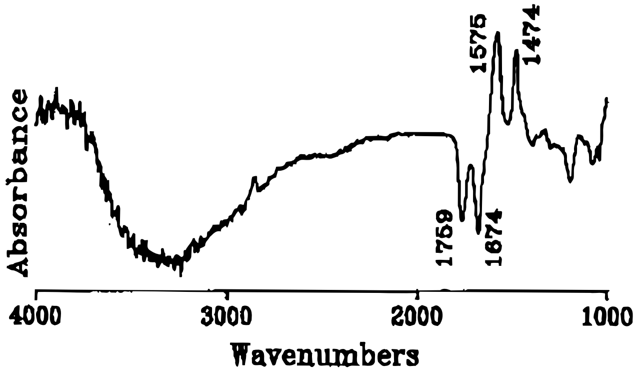


Figure 5. Difference spectra between two desorption steps in addition of NHP (to be compared with Figure 4): from room temperature to 423 K.

Because of the hydrogen bonding and the N-coordination, the overall adsorption of NHP is more complex than that of the NMP. Evidently, it has a strong association on the surface at room temperature. However, the thermal stability is poor, due to the following observation: (i) the carbonyl band at 1776 cm^{-1} disappeared as shown in the difference spectrum between the two steps (Figure 5), which suggests the complete dissociation of the N-coordination by NHP molecules; (ii) the hydrogen bonding appeared to be very weak at 423 K (Figure 4b), only one-fifth of its original intensity of the room-temperature spectrum remained.

Conclusion

The surface reactions of nanostructured AlN powder with two cyclic amides, NMP and NHP, were studied by FT-IR spectroscopy. The reactivity and adsorption characteristics of these surface interactions are summarized as follows: (i) Strong hydrogen bonding was observed in the adsorption of NMP on the AlN surface. The unusual thermal stability of this hydrogen bond was evident as the system temperature increased to 423 K, which was interpreted as a secondary interaction of the N-alkylated amide with the surface acidic Al^{3+} sites. (ii) Careful analyses of the NHP adsorption at room temperature revealed a synergistic effect originating

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from both the complex coordination by the amide nitrogen with the surface Lewis site and the surface hydrogen bonding. The complex coordination is of particular interest since it proved, for the first time, a unique surface characteristic of the chemically synthesized AlN nanopowders. (iii) The proton-transfer mechanism involved in the formation of the complex coordination is of significance to the deagglomeration and stabilization process for the ceramic nanoparticles. These proton-transfer processes would be responsible for the formation of an ionic double layer which changes the surface acid−base character. If the concentration of the dispersant dissolved in the organic phase is sufficiently high, then dynamic adsorption and desorption occur, and some proton-carrying dispersant will desorb into the suspension and provide counterions for the charges remaining on the surface. This explains why acid−base interactions act very effectively as suspension stabilization forces. Notice that the formation of this active surface adsorption is particularly associated with the nanoparticle suspension or nanoscale structures. As a nanoparticle possesses a large surface-to-volume ratio, the number of surface sites and the overall surface reactivity dramatically increase as compared to the adsorption on micron-sized particles.

Finally, it should be recognized that the strength and thermal stability of the surface hydrogen bonding with NMP are of importance to the properties of AlN suspensions, since the processing for the nanopowders involved a sonication step that created an extensive amount of heat during the treatment. Moreover, the surface adsorption pattern with NHP can be used to estimate the ability of the ceramic powders to interact with the unsubstituted amide and as a model compound for more complex substances such as poly(amic acid), the precursor polymer to polyimide.

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