

**NASA
Technical
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2449**

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March 1985

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Humidity Effects on Adhesion
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Summary

The effects of a moist nitrogen environment on the adhesion of Ni-Zn ferrite and magnetic tape in elastic contact with a Ni-Zn ferrite hemispherical pin were investigated at loads to approximately 1.6 mN in dry, humid, and saturated nitrogen at 23 °C. Adhesion was found to be independent of normal load. Ferrites adhere to ferrites in a saturated atmosphere primarily from the surface tension effects of a thin film of water adsorbed on the ferrite surfaces. The surface tension of the water film calculated from the adhesion results was 48×10^{-5} to 56×10^{-5} N/cm; the accepted value for water is 72.7×10^{-5} N/cm. The adhesion of ferrite-ferrite contacts increased gradually with increases in relative humidity to 80 percent but rose rapidly above 80 percent. The adhesion at saturation was 30 times or more greater than that below 80 percent relative humidity. Although the adhesion of magnetic tape-ferrite contacts remained low below 40 percent relative humidity and the effect of humidity on adhesion was small, the adhesion increased greatly with increasing relative humidity above 40 percent. Changes in the adhesion of elastic contacts were reversible on humidifying and dehumidifying.

Introduction

Recording devices, both audio and video, and computer systems are used extensively in space vehicles such as the shuttle and in ever-increasing numbers in advanced aircraft. Greater reliability in these components is being demanded.

Conventional magnetic recording is accomplished by the relative motion of a magnetic tape in sliding or intermittent contact with a stationary (audio or computer) or rotating (video) read/write magnetic head in order to achieve high density and high resolution. High adhesion and friction and a small amount of magnetic head and tape wear may render the recording process unreliable. Much research has been conducted into friction and wear properties of magnetic heads and tapes (refs. 1 to 7). Their adhesion properties have not as yet been studied in great enough detail (ref. 8).

The sensitive wire-fulcrum type of adhesion apparatus used in this investigation was developed by Masuo and

Maeda and their group (ref. 9). With this apparatus adhesion as small as 1 μ N can be measured.

This investigation examined the effects of humidity (water vapor) on the adhesion of Ni-Zn ferrite in elastic contact with magnetic tape and itself at loads to approximately 1.6 mN in dry, moist, and saturated nitrogen.

Background

The environment significantly changes the surface interactions between magnetic tapes and solids (refs. 1, 8, and 10 to 15). Carroll and Gotham (ref. 1) briefly state that the friction and abrasiveness of magnetic tape depend on humidity. Bradshaw and Bhushan (ref. 8) indicate that the coefficient of friction increases with brief increases in humidity even for tapes of markedly different composition and conditions of manufacture. Long periods (several weeks) of exposure to a humid environment at elevated temperatures can dramatically change the frictional behavior of a magnetic tape as a result of hydrolytic degradation. Miyoshi and Buckley (refs. 10 and 11) indicate that frictional behavior responds quickly (of the order of seconds) not only to humid exposure, but also to dry exposure. The friction responses during humidifying and dehumidifying are similar (fig. 1) and reversible in both elastic and plastic contacts. Surface softening of magnetic tape due to water vapor is one of the mechanisms important to humidity effects on frictional behavior. Although friction results have demonstrated clearly the existence of a humidity effect, the adhesion between magnetic tape and a solid is not fully understood.

Materials

The magnetic tape used in this investigation had a layered structure: a magnetic layer of CrO₂ powders, binder, and lubricant; a polymer base film; and a back-coating layer (fig. 2). It was 23 μ m thick and 12.7 mm wide. The magnetic layer resembled emery, a familiar abrasive. The composition, surface roughness, and Knoop hardness of the tape are presented in table I.

The hot-pressed, polycrystalline Ni-Zn ferrite is a ceramic semiconductor. The Ni-Zn ferrite hemispherical

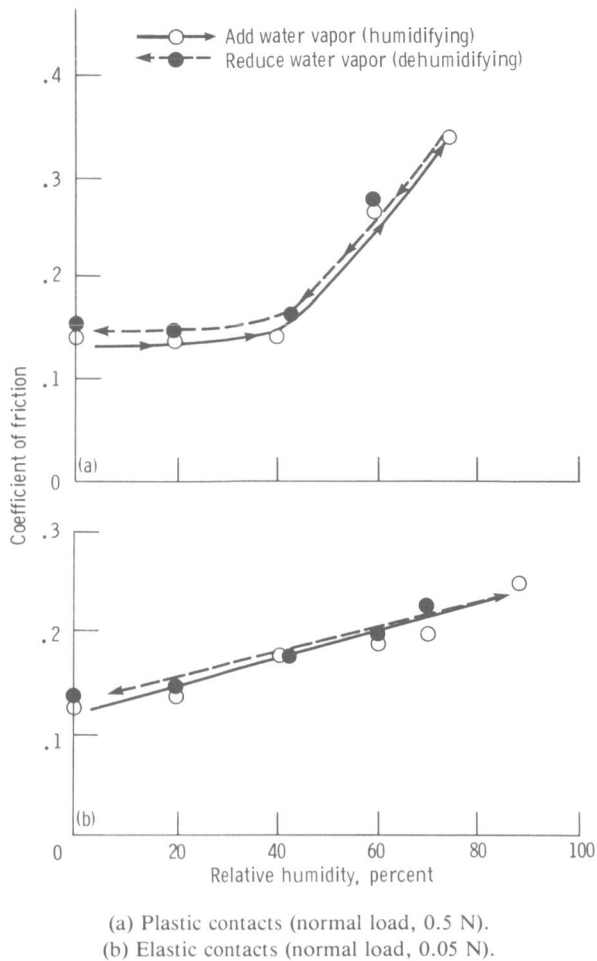
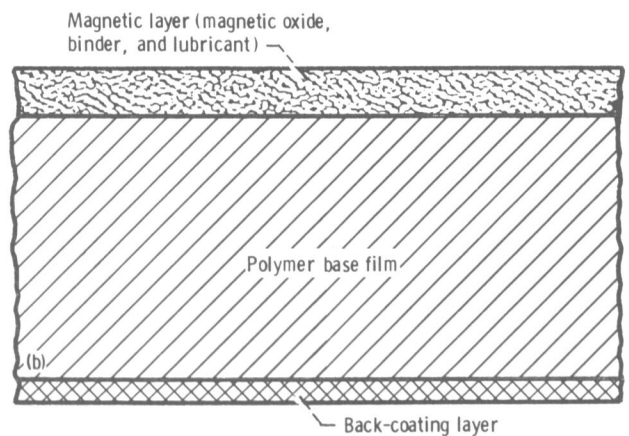
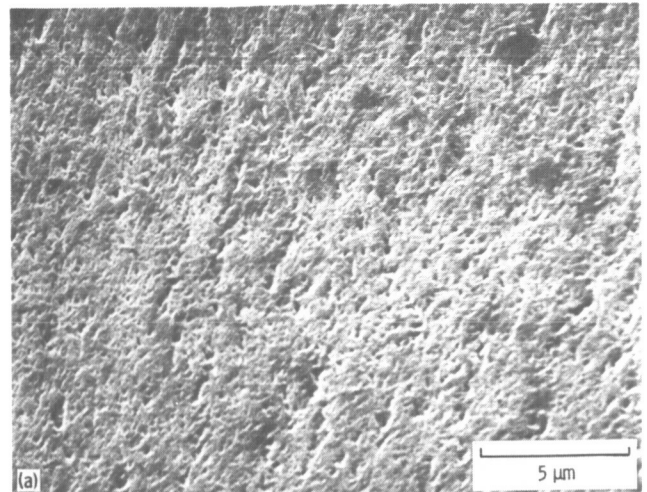


Figure 1.—Effect of humidifying and dehumidifying on adhesion of magnetic tape in contact with Ni-Zn ferrite pin (ref. 11). Sliding velocity, 0.1 mm/s; temperature, 23 °C; atmosphere, nitrogen.

pin (radius, 2 mm) and flat specimens were polished with diamond powders (particle diameter, 3 and 1 μm) and with 1- μm Al_2O_3 powder. The polished surface of Ni-Zn ferrite, examined by optical microscope using Nomarski differential interference contrast, typically contained scratches and asperities (fig. 3). The surface roughness, measured by a profilometer, was 0.1 μm for the maximum height of irregularities (table II). The surface chemically etched with an HCl solution at 50 °C contained grain boundaries, pores, and scratches (fig. 4). Grain size was obtained by averaging the measurements of 50 grains or more in scanning electron photomicrographs. The grain size of Ni-Zn ferrite was about 8 μm (ref. 16), and the porosity was less than 0.1 percent. The composition, grain size, porosity, Vickers hardness, and surface roughness of Ni-Zn ferrite are summarized in table II.



(a) Scanning electron micrograph.
(b) Schematic.

Figure 2.—Magnetic tape.

TABLE I.—COMPOSITION AND PROPERTIES OF MAGNETIC TAPE

Magnetic particles	CrO_2
Particle loading: ^a	
Percent by volume	50
Percent by weight	80
Binder	Nitrocellulose, polyester-polyurethane, and polyurethane (hard component)
Lubricant	Fatty acid ester
Base film	Polyethylene terephthalate
Surface roughness, ^b nm	27.3
Knoop hardness ^c at 13 °C, MPa	178

^aMagnetic particle concentration.

^bRoot-mean-square roughness.

^cMeasuring load, 1.3 mN.

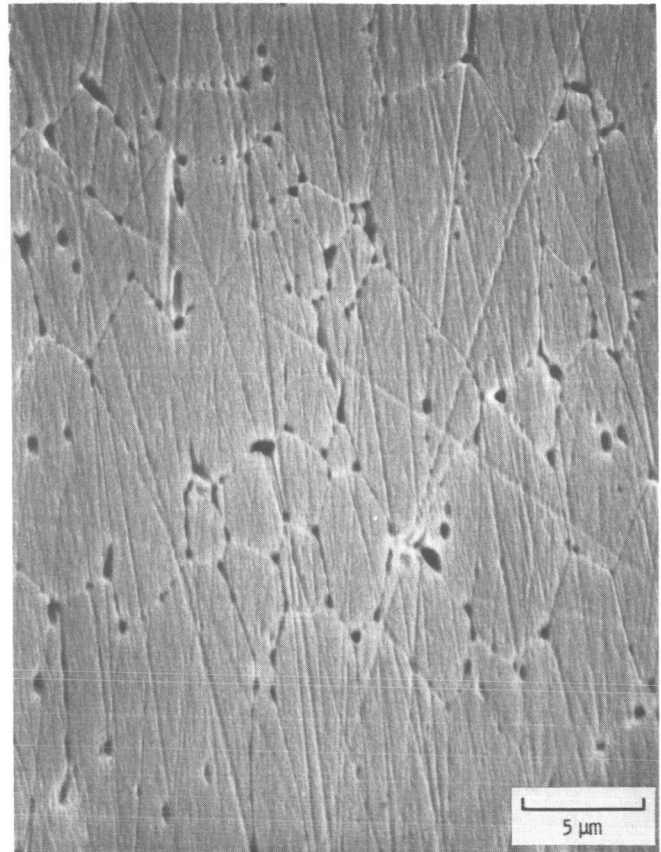
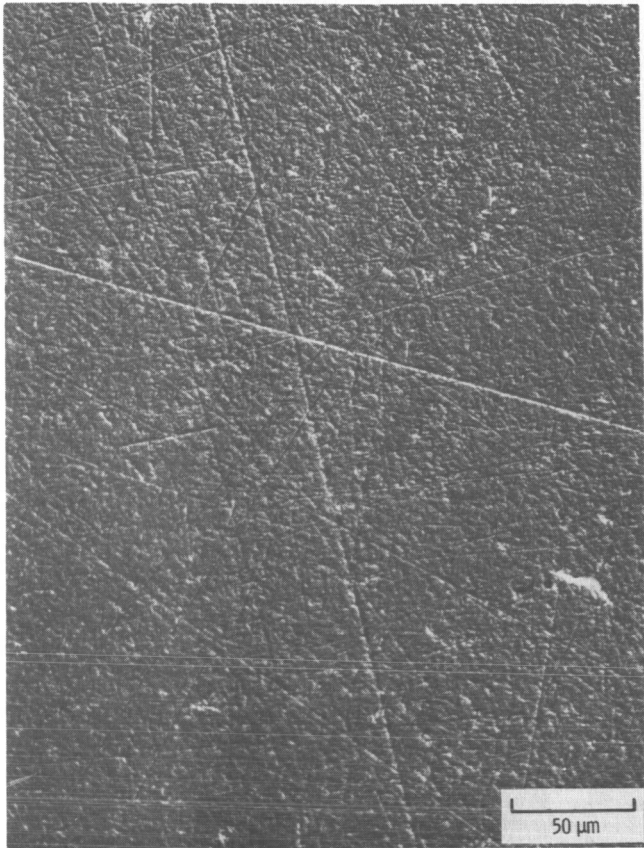


Figure 3.—Optical photomicrograph of polished Ni-Zn ferrite surface taken under reflected light using Nomarski differential interference contrast.

Figure 4.—Scanning electron photomicrograph of chemically etched Ni-Zn ferrite surface.

TABLE II.—COMPOSITION AND HARDNESS OF HOT-PRESSED POLYCRYSTALLINE Ni-Zn FERRITE

Composition, wt%	66.6Fe ₂ O ₃ -11.1NiO-22.2ZnO
Grain size, μm	8
Porosity, percent	<0.1
Vickers hardness ^a	715
Surface roughness ^b , μm	0.1

^aMeasuring load, 0.5 N.

^bMaximum height of irregularities, as measured by surface profilometer.

Apparatus

The apparatus used in this investigation (fig. 5) was based on a torsion mechanism. It was basically a pin on a flat. The Ni-Zn ferrite flat or the magnetic tape was mounted on a stainless steel support and retained on a micrometer-head-screw-driven platform moved either by an electric motor or manually. The ferrite pin was mounted on one end of a movable beam. A free-moving, rod-shaped magnetic core was mounted on the other end of the beam. The coils of a linear variable differential transformer were mounted on a stationary beam. There was no physical contact between the movable magnetic core and the coil structure. The movable beam was supported by a torsion (music) wire.

The flat ferrite or magnetic tape specimen was moved toward the ferrite pin, pressed against it with a known force, and then moved back horizontally until the pin and

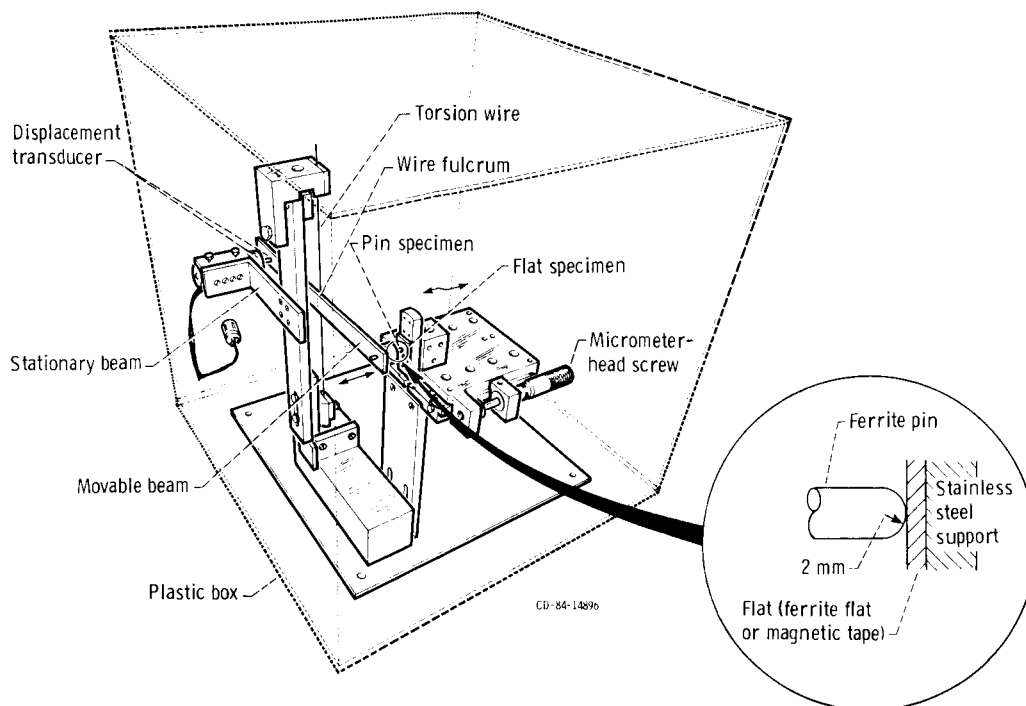


Figure 5.—Apparatus for measuring adhesion.

flat were pulled apart. The displacement of the pin specimen provided a measure of the applied normal load or adhesion force. The adhesion force was the force required to separate the pin and flat surfaces in the normal direction. The entire apparatus was housed in a plastic box, which was filled with dry or humid nitrogen.

Experimental Procedure

When the apparatus was being calibrated, the movable beam was placed in the horizontal position, and then a first-class standard weight to 1.96 mN was placed on a weight support plate located at the pin specimen. Deflection of the movable beam, as monitored by the linear variable differential transformer, was proportional to the load.

The polished Ni-Zn ferrite pin and flat specimens were rinsed with 200 or 190 proof ethyl alcohol. After dry nitrogen was admitted into the system (fig. 5), a polished Ni-Zn ferrite pin and a polished Ni-Zn ferrite flat or an as-received magnetic tape were placed in the experimental apparatus and maintained in dry nitrogen for 15 min. The specimen surfaces were then brought into contact by moving the micrometer-head screw forward either manually or by an electric motor (0.8 rpm) and loaded.

To obtain consistent experimental conditions, contact was maintained for 30 s and then the flat specimen was pulled apart from the pin specimen by gradually moving

the micrometer-head screw backward. The atmosphere was then humidified to the desired relative humidity of 80 percent by admitting humid nitrogen into the system. After further adhesion experiments the system was gradually dehumidified to a dry nitrogen atmosphere. Adhesion experiments were also conducted at humidities from 80 percent relative to saturation.

Adhesion experiments were conducted on different locations of the same specimen in the different atmospheres. Both the load and the adhesion force were monitored by the linear variable differential transformer.

On a typical force-time trace resulting from such adhesion experiments (fig. 6) contact occurs at point A. The line A-B represents the region where the load is being applied. The line B-C represents the region where the contact is maintained at a given load and the surfaces of the specimens are stationary. Line C-D represents the region where both the unloading and separation forces are being applied on the adhered junction. At point D the onset of separation occurs. After separation of the flat specimen from the pin, the pin fluctuates back and forth.

Each adhesion force value is the average from 10 or more experiments for the manually moved micrometer-head screw and the average of three or more experiments for the motor-driven screw. Humidity was measured by a sensor using the capacitance change in a thin-film polymer capacitor. The capacitor reacted quickly and gave a very short response time (e.g., typical response time for 90 percent response was 5 s). The accuracy of

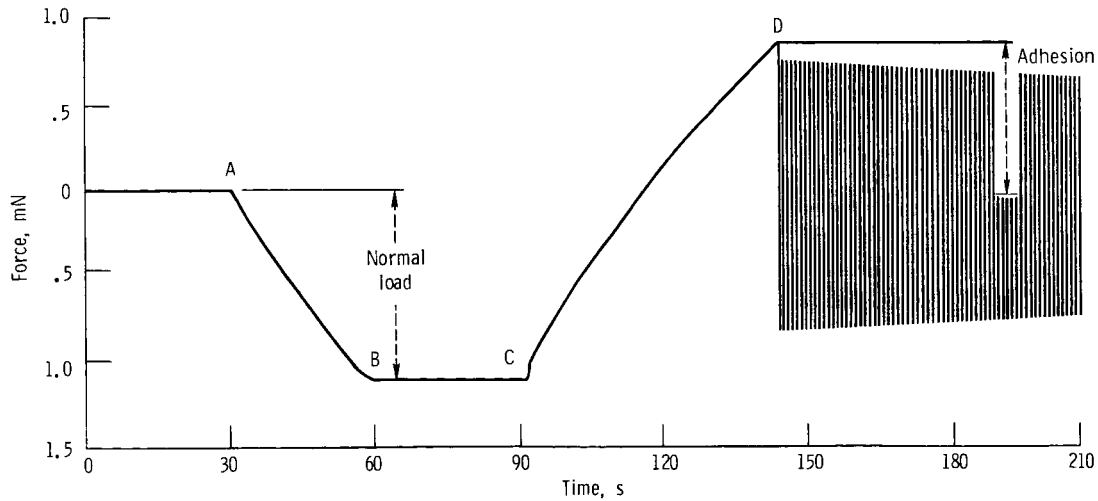


Figure 6.—Typical force-time trace—a Ni-Zn ferrite pin in contact with a Ni-Zn ferrite flat in an almost-saturated nitrogen atmosphere.

this sensor system at 20 °C is approximately ± 2 percent from 0 to 80 percent relative humidity and ± 3 percent from 80 to 100 percent. Humidity calibration of the system was based on the known equilibrium relative humidity of saturated salt solutions. The humidity probe was placed in the plastic box shown in figure 5.

Results and Discussion

Effect of Water Films

The adhesion forces on a ferrite pin in a saturated nitrogen atmosphere (fig. 7) were high, constant, and independent of the normal load. It is natural to expect that the adhesion observed arose primarily from the surface tension effects of a thin film of water adsorbed on the ferrite surfaces. The happenings at the interface may be visualized as follows: a ferrite pin is essentially in elastic contact with a flat ferrite surface, with a thin film of water between them (fig. 8(a)). When the normal load is removed and the elastic stresses within the bulk of the specimens are released, the junctions are broken one by one. When the pin (of radius R) detaches from the flat (fig. 8(b)), the applied separation force is balanced by the surface tension of a thin film of water resisting the extension of the surface. Suppose the liquid collects to form a pool at the tip of the hemispherical pin and the radius of curvature of the profile of the meniscus is r . If the meniscus is very small ($r \ll R$) and the liquid completely wets the surface (i.e., the contact angle is zero), the pressure p inside the liquid is less than atmospheric pressure by approximately T/r , where T is the surface tension of the liquid. This acts over an area πa^2 of the water pool, giving a total adhesive force of $p\pi a^2$ (i.e., $\pi a^2 T/r$). To a close approximation $a^2 = 2R \times 2r$, where R is the radius of curvature of the spherical surface. The resulting adhesive force is

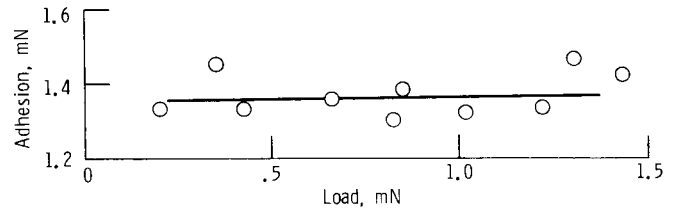


Figure 7.—Adhesion as a function of normal load for a Ni-Zn ferrite pin in contact with a Ni-Zn ferrite flat in a saturated nitrogen atmosphere. Room temperature.

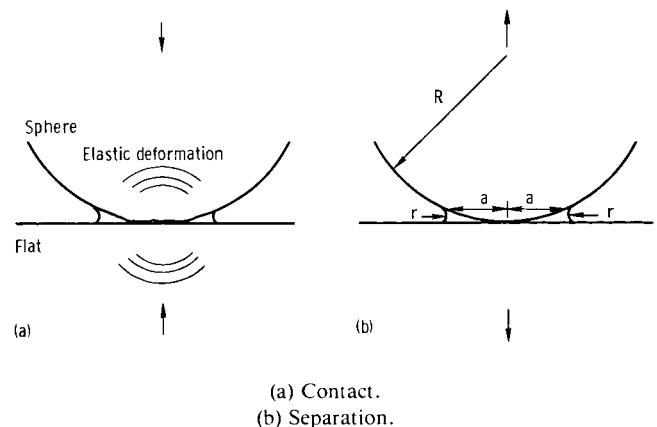


Figure 8.—Meniscus formed at contact area between a spherical surface and a flat surface. (Modified from refs. 17 and 18.)

$$Z = 4Rr\pi \left(\frac{T}{r} \right) = 4\pi RT \quad (1)$$

(refs. 17 to 19).

Adhesion was thus independent both of the thickness of the water film and the applied normal load. The surface tension calculated from these results (fig. 7) was 48×10^{-5} to 56×10^{-5} N/cm; the accepted value for water is 72.7×10^{-5} N/cm. This discrepancy may be due to the surface roughness and irregularities of roundness or flatness of the ferrite specimens (fig. 3). The irregularities can affect the radii of curvature of the hemispherical pin and the meniscus.

Effect of Humidity

Ferrite-ferrite contacts.—For a Ni-Zn ferrite pin in elastic contact with a Ni-Zn ferrite flat in dry and humid nitrogen (60 percent RH) (fig. 9), there was no change in adhesion with normal load in either atmosphere, but adhesion was higher at 60 percent relative humidity. Similar experiments were therefore conducted at various relative humidities (fig. 10). Adhesion increased continuously with increasing relative humidity to 80 percent but rose rapidly above 80 percent. At approximately 87 percent relative humidity and in saturated nitrogen, adhesion was extremely high because of the great surface tension effects of thin water films adsorbed on the ferrite surfaces.

Magnetic tape-ferrite contacts.—A Ni-Zn ferrite pin in elastic contact with a magnetic tape in dry and 60-percent-relative-humidity nitrogen (fig. 11) also showed no effect of normal load on adhesion and higher adhesion at 60 percent relative humidity. Similar experiments were therefore conducted to examine the humidity effect at loads of 0.67 to 0.87 mN (fig. 12). In dry nitrogen adhesion was $17 \mu\text{N}$ at a load of 0.87 mN. The humidity was then raised to 80 percent by admitting humid nitrogen into the system. The adhesion increased

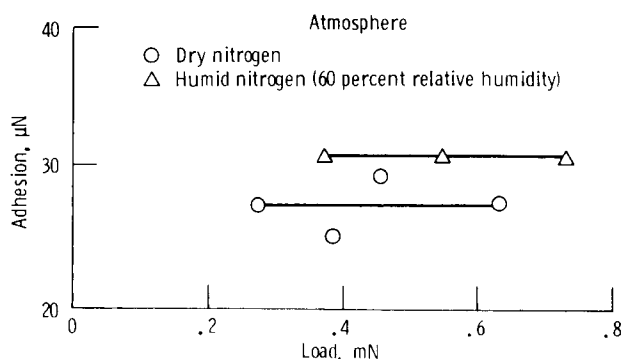


Figure 9.—Adhesion as a function of normal load for a Ni-Zn ferrite pin in contact with a Ni-Zn ferrite flat in dry and humid nitrogen atmospheres.

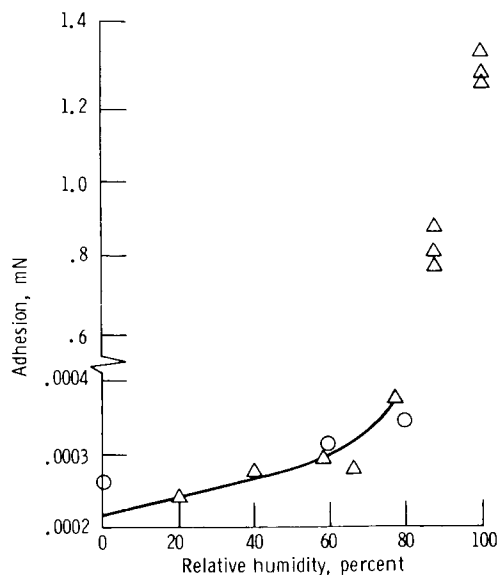


Figure 10.—Adhesion as a function of humidity for a Ni-Zn ferrite pin in elastic contact with a Ni-Zn ferrite flat in nitrogen atmosphere.

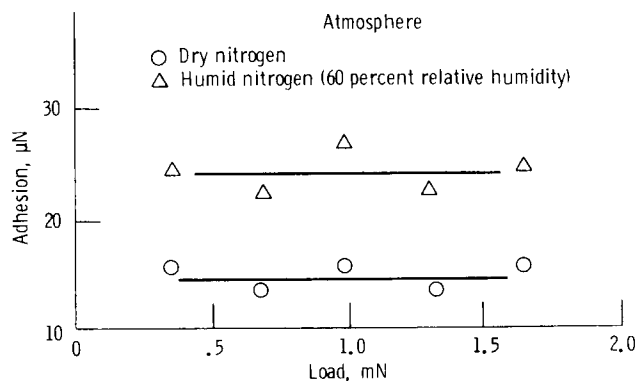


Figure 11.—Adhesion as function of normal load for magnetic tape in contact with a Ni-Zn ferrite pin in dry and humid nitrogen atmospheres.

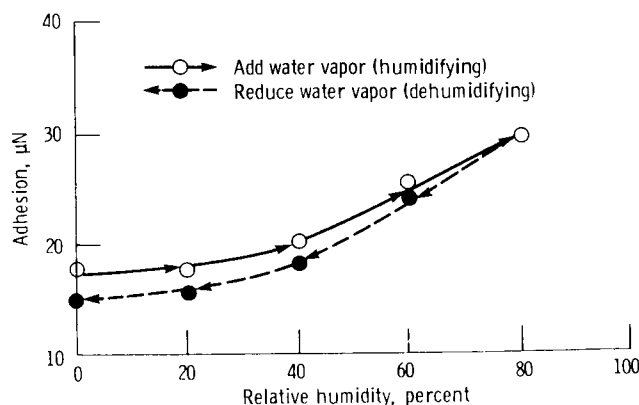


Figure 12.—Effect of humidifying and dehumidifying on adhesion of magnetic tape to Ni-Zn ferrite pin. Load, 0.67 to 0.87 mN.

slightly below 40 percent relative humidity and greatly above that value. On dehumidifying, the adhesion decreased with decreasing relative humidity in a similar manner. The changes in adhesion on humidifying and dehumidifying are therefore reversible.

Adsorption of water vapor changes the interaction forces and the chemistry of the tape at the interface. The surface tension of a thin film of water adsorbed on the magnetic tape-ferrite or ferrite-ferrite contact is a primary mechanism of adhesion during short exposure to a humid atmosphere.

Conclusions

From adhesion experiments conducted with a Ni-Zn ferrite flat and a magnetic tape in elastic contact with a Ni-Zn ferrite hemispherical pin in dry, humid, and saturated nitrogen, the following conclusions were drawn:

1. Adhesion is independent of the normal load.
2. The adhesion of ferrite-ferrite contacts in a saturated atmosphere arises primarily from the surface tension effects of a thin film of water adsorbed on the surfaces. The surface tension of the water film was calculated as 48×10^{-5} to 56×10^{-5} N/cm; the accepted value for water is 72.7×10^{-5} N/cm.
3. The adhesion of ferrite-ferrite contacts increases gradually with increases in relative humidity to 80 percent but rises rapidly above that value. The adhesion at saturation is 30 times or more greater than that below 80 percent relative humidity.
4. Although the adhesion of magnetic tape-ferrite contacts remains low below 40 percent relative humidity and the effect of humidity was small, the adhesion increased greatly with increasing relative humidity above 40 percent. The changes in adhesion in elastic contacts are reversible on humidifying and dehumidifying.

National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio, January 15, 1985

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1. Report No. NASA TP- 2449		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle Humidity Effects on Adhesion of Nickel-Zinc Ferrite in Elastic Contact With Magnetic Tape and Itself				5. Report Date March 1985	
				6. Performing Organization Code 506-53-1B	
7. Author(s) Kazuhisa Miyoshi, Donald H. Buckley, Tomohide Kusaka, and Chikayoshi Maeda				8. Performing Organization Report No. E-2361	
				10. Work Unit No.	
9. Performing Organization Name and Address National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135				11. Contract or Grant No.	
				13. Type of Report and Period Covered Technical Paper	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546				14. Sponsoring Agency Code	
15. Supplementary Notes Kazuhisa Miyoshi and Donald H. Buckley, Lewis Research Center; Tomohide Kusaka and Chikayoshi Maeda, Osaka Institute of Technology, Osaka, Japan.					
16. Abstract The effects of humidity on the adhesion of Ni-Zn ferrite and magnetic tape in elastic contact with a Ni-Zn ferrite hemispherical pin in moist nitrogen were studied. Adhesion was independent of normal load in dry, humid, and saturated nitrogen. Ferrites adhere to ferrites in a saturated atmosphere primarily from the surface tension effects of a thin film of water adsorbed on the ferrite surfaces. The surface tension of the water film calculated from the adhesion results was 48×10^{-5} to 56×10^{-5} N/cm; the accepted value for water is 72.7×10^{-5} N/cm. The adhesion of ferrite-ferrite contacts increased gradually with increases in relative humidity to 80 percent but rose rapidly above 80 percent. The adhesion at saturation was 30 times or more greater than that below 80 percent relative humidity. Although the adhesion of magnetic tape - ferrite contacts remained low below 40 percent relative humidity and the effect of humidity was small, the adhesion increased considerably with increasing relative humidity above 40 percent. The changes in adhesion of elastic contacts were reversible on humidifying and dehumidifying.					
17. Key Words (Suggested by Author(s)) Ferrite Adhesion Magnetic tape Humidity			18. Distribution Statement Unclassified - unlimited STAR Category 27		
19. Security Classif. (of this report) Unclassified		20. Security Classif. (of this page) Unclassified		21. No. of pages 9	22. Price* A02

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