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Use of Atomic Oxygen for the Determination of Document Alteration

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September 2003

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Abstract

Atomic oxygen, which normally is found only the near Earth space environment, causes oxidation and erosion of polymers on spacecraft. The development of technology to prevent this degradation has required NASA to develop ground laboratory facilities that generate atomic oxygen. Atomic oxygen has also been found to be able to oxidize most types of ink from a variety of types of pens. The use of atomic oxygen to identify alteration of documents has been investigated and is reported. Results of testing indicates that for many types of ink, pens, and paper, identification of document alteration of pen and ink numbers and evidence of alteration can be made visible by exposing the questionable writing to atomic oxygen. Atomic oxygen provides discrimination because different inks may oxidize at different rates, the amount of time between delayed alteration may add to ink thickness at crossings, and the end of pen strokes tends to have much thicker ink deposits than the rest of the character. Examples and techniques of using atomic oxygen to identify document alteration indicate that the technology can, in many but not all cases, provide discrimination between original and altered documents.

Introduction

Atomic oxygen is produced in the near Earth space environment by short wavelength solar radiation causing scission of the diatomic oxygen bonds. Recombination with other atomic oxygen atoms, the formation of ozone, or the reaction with diatomic nitrogen does not readily occur because the mean free path is so large that reaction probabilities are extremely low at the altitudes between 180 and 650 km where atomic oxygen is the dominant species (ref. 1). Atomic oxygen is able to react with most spacecraft polymers causing gradual oxidative thinning. To protect spacecraft polymers from thinning, NASA and other aerospace organizations have had to develop atomic oxygen protective coatings and demonstrate their effectiveness in preventing atomic oxygen degradation by constructing facilities that produce atomic oxygen in the laboratory (refs. 2 to 6).

As a result of becoming familiar with the ability of atomic oxygen to oxidize hydrocarbon polymers and carbon, many spin-off applications of atomic oxygen have been discovered and investigated including using atomic oxygen to clean soot and char off the surfaces of fire damaged paintings (refs. 7 to 11) as well as potential biomedical applications of atomic oxygen textured surfaces (ref. 12).

The concept of using atomic oxygen to determine if a written document is altered was conceived by Lynda Taylor-Hartwick of the Independent Association of Questioned Document Examiners, Inc. (IAQDE) after reading an article concerning use of atomic oxygen for art restoration. Her concept was then brought to the authors' attention. The concept appeared feasible in that not all inks are identical and the atomic oxygen oxidation of ink may cause altered pen marks to look differently than the original pen marks. As a result of the apparent feasibility of using atomic oxygen to discriminate between original and altered pen characters, an investigation was conducted in collaboration with the Independent Association of Questioned Document Examiners, Inc.

The premise that alteration of pen and ink written numbers could be detected by exposure of samples to atomic oxygen was based on the following rationale: different inks may oxidize at different rates, alterations made after the ink has had time to dry may add to ink thickness at crossings and lastly, the end of pen strokes have thicker deposits because the pen comes to a stop or near stop there.

Apparatus and Procedure

Samples of blue and black pens of ballpoint, felt tip, gel, and roller ball types were provided by the IAQDE and NASA to test the atomic oxygen interaction with various types and colors of pens and ink (see table I). Most of the pen marks were tested by writing on check stock (see fig. 1) also supplied by the IAQDE. However, a variety of texture (smooth to porous surface) papers of unknown origin were also used to assess the dependence of atomic oxygen exposure results for various pens and ink on the type of paper used.

Atomic oxygen testing was performed on samples that were placed in a SPI Plasma Prep II 13.56 MHz radio frequency plasma asher operated on air at a pressure of ~ 80 millitorr. The asher was operated at a Kapton effective flux of 4.69×10^{15} atoms/(cm^2sec) (ref. 13). Typical durations of exposure of ink samples on check stock were 50 minutes, which was a Kapton effective fluence of 1.41×10^{19} atoms/cm^2. Ink marks were made on small (~ 1.5 cm × 3 cm) pieces of check stock and placed on a glass tray, as shown in figure 2(a), then inserted in the plasma asher to expose only the ink marked side to atomic oxygen (fig 2(b)).

Because the majority of the tests were performed on small pieces of paper cut from checks, a device needed to be created to expose only a portion of a document without cutting apart an entire document. One method of obtaining this goal was to protect the majority of the document that was not to be exposed to the atomic oxygen with a polymer covering such as Kapton with a hole cut into the covering over the questionable area. The entire check was wrapped around a small glass bottle (16 cm high, 5 cm diameter) and a thin piece of Kapton was taped in place over it, with a small window cut into the Kapton in order to expose the suspicious ink markings (see fig. 3(a)). The bottle was inserted into the glass cylinder of the plasma asher in such a manner that the unmasked area would be exposed to atomic oxygen (see fig. 3(b)). The atomic oxygen would then only react with the exposed area of the check and leave the rest of the check untouched. However, this method of exposing only a small portion of a document without cutting it apart could only work on smaller documents, such as checks (~ 7 cm × 15.5 cm), in order for them to fit in the plasma asher.

A test of atomic oxygen exposure of ink samples on a full sheet of 8.5 inch × 11 inch paper was also conducted using an atmospheric atomic oxygen beam (refs. 10 and 11). This atomic oxygen exposure system operates in air and is useful for large documents. Documents can remain flat with only a small area of the surface exposed to the atomic oxygen beam (2-3 mm in diameter). Figure 4(a) shows the overall atmospheric atomic oxygen system. Figure 4(b) shows how a sample of paper can simply be held up to expose it to the atmospheric atomic oxygen beam. Close-up photographs of the atmospheric atomic oxygen beam and nozzle are shown in figure 4(c). As can be seen, the beam is very narrow and can thus be directed to treat a specific character or part of a character being examined.

Procedure to determine if different inks may oxidize at different rates.

A matrix was used consisting of an array of "X" ink marks, as shown in figure 5, where one line making up the "X" was from a different pen of the same color. The sample was then photographed with a digital camera through an Olympus microscope prior to and after atomic oxygen exposure in the plasma asher.

Procedure to determine if alterations made after the ink dries add to ink thickness at crossings.

The concept for this determination can be most easily visualized if one considers making an "X" using a paint brush in which you make both line strokes prior to the paint (or ink) drying for an unaltered number. This would be compared with an altered document in which the paint for the first line of the "X" would be dry prior to adding the second line that completes the "X". In the later case the paint (or ink) would be double the thickness at the crossing when compared to the former case as illustrated in figure 6. Digital photos were taken prior to and after atomic oxygen exposure of "X's" made with and without delay in making the individual lines that make up the character. A delay time of one hour was used to assure that the ink was dry for the delayed alterations. The same type of "X" matrix was used for these tests as for the tests to compare the oxidation rates of different inks. An assessment of how dark the crossing region of the "X" was after atomic oxygen exposure compared to the straight-line non-crossing areas was used to determine the merits of using this concept for identification of document alteration. This was performed using the digital camera through an Olympus microscope.

Procedure to determine if the ends of pen strokes have thicker deposits than the rest of the pen stroke.

Because fast pen movement across paper tends to place a thinner ink deposit than slow pen movement, the termination of a stroke or character would produce a much thicker ink deposit than the intermediate or start of the pen strokes. Thus simply exposing unaltered and altered numbers to atomic oxygen should show dark spots at the end of strokes which differ depending upon whether or not the number was altered. Microscopic comparison using a digital camera through an Olympus microscope was performed before and after atomic oxygen exposure to assess the effectiveness of using this technique for identification of document alteration.

Results and Discussion

Different inks may oxidize at different rates.

All inks do not have the same chemical composition, thus the thickness and atomic oxygen erosion rate of the inks would be expected to be different. On documents that are altered it is probable that the inks used will come from different brands and types of pens, which can result in the altered ink strokes oxidizing at a different rate than the original ink. As many of the original and altered ink strokes were oxidized and became visibly lighter, the altered ink often appeared lighter or darker than the original ink. In some cases, the type of pen determined what area of the stroke would oxidize and fade the fastest. Many of the pens in the test matrix of table I appeared to oxidize at similar rates with one another, but different qualities of the strokes became apparent, depending on what type of pen the stroke was written with.

Five different felt tip pen inks were also exposed to atomic oxygen in order to determine whether different inks written by the same type of pen (but different brands) would oxidize at the same rate. All of the ink strokes were initially the same shade of black prior to atomic oxygen exposure. Although the ink samples all had similar characteristics in their strokes such as thick ink accumulation at the end of a stroke and the uniform oxidation throughout the line, the oxidation of some of the inks occurred at different rates allowing discrimination between inks. Also, different colors of the inks of the felt tip pen ink were revealed when the samples were exposed. Some shades of green and gold appeared intertwined with the black in some of the inks in varying degrees, while the ink of the Paper Mate felt tip pen remained very black. As can be seen in figure 7, the unknown fine point pen appears to have oxidized the most, since the entire stroke, excluding the ink accumulation at the end of the stroke, has been almost entirely removed from the paper. The other inks were not as faded as the unknown pen ink, but there are some subtle differences to how the strokes oxidized. For example, the ink accumulation marks at the end of the stroke are much more defined for the ink from the Expresso Medium Felt Tip Pen than the rest of the pens. Also, as an unexpected result, the color change of some of the various inks created a definite visual distinction that can be used to help identify the different inks.

It was found that Sharpie Permanent Marker does not become oxidized when exposed to atomic oxygen (probably due to pigment other than carbon or hydrocarbon). Other inks, particularly the felt tip pen ink and the roller ball pen ink, are able to write a stroke that appears very similar to the ink written by the permanent marker, but these inks are able to be oxidized by atomic oxygen and, therefore, removed from paper. A single written number was constructed in which a portion of the number was written with the felt tip pen ink (atomic oxygen removable) and the other piece was written with the Sharpie permanent marker (see figs 8(a) and (b)). The inks of the different pens looked identical, and it seemed that the number had been constructed with the same pen. Once the number was exposed to atomic oxygen (in this case using the atmospheric atomic oxygen beam which was impinged on the first digit of the number for a couple minutes), it became apparent that the number had been written with two different inks (see fig. 8(c)).

The inks from the felt tip pen had been oxidized by the atomic oxygen and had been drastically removed from the paper. This ink, being extremely lighter than before, made a great contrast with the ink from the permanent marker, which remained very dark due to its inability to be oxidized. It was clear that the number had been constructed out of two different inks, which is a definite sign that a number had been altered. The probability that a person would purposely write a number with two different types of pens is very low, whereas the probability that an alteration would be written with ink that is different from the original ink is very high.

Alterations made after the ink dries add to ink thickness at crossings.

Delayed alteration of a stroke may cause the ink to pile up at an intersection, requiring a greater quantity of time in order to be oxidized when exposed to atomic oxygen. The contrast between the layered ink at the crossing and the thinner areas becomes more apparent the longer the ink is exposed to atomic oxygen. The layered ink at the crossing takes a longer period of time to oxidize and be removed from the paper than the ink around the crossing does, since it is thinner. As a result, the crossing appears to be much darker than the rest of the ink around it.

For the crosses written with or without delay for the felt, gel, and roller ball pens, no visual difference was noted at the crossings between a crossing written with without delay.. The ink at the crossings was uniform with all of the ink around it on both crosses (with and without delay), so the dark spots that result from the pile up of ink at the crossing are not yet visible. Using visual observation, there was no discriminating factors that revealed which of the crosses was written with a delay and which of the crosses was written without one. However, the ink did in fact often layer at the crossing, so the atomic oxygen is required to show the difference by oxidizing the thicker crossing and the ink around this crossing, which is much thinner. The crossing remained much thicker than the rest of the ink, creating the appearance that the intersection is much darker than the rest of the ink of the “X” (see fig. 6). The crossings of the ballpoint pen without the delay already were darker than the rest of the ink, so they did not follow the same type of pattern as the other pens. However, some of the crossings with the delay would be significantly darker than the crossings without the delay. The difference in shade of the crossings with and without the delay for the ballpoint pen would be much subtler than that of the other pen types, but it was still present.

In some circumstances, the results from the experiment contradicted the expected theory. The crossing of the “X” written without the delay actually appeared darker than the crossing written with the delay, implying that the intersection was thickest at the crossing in which the two strokes were applied one immediately after the other (see fig. 9). These results create a direct contradiction with the idea of the layering of the ink. The instances in which this occurred were not common, but this suggests that atomic oxygen will not successfully discriminate, in all circumstances, between strokes made with and without delays in crossings.

The ends of pen strokes have thicker deposits than the rest of the pen stroke.

It takes longer for thicker layers to be oxidized by the atomic oxygen, so if the thicker area is surrounded by thinner layers, the thinner layers will be removed entirely before the thicker layers can be. As a result, the thicker areas will remain darker longer when exposed to atomic oxygen as compared to the thinner areas. Since the end of the pen strokes tend to have thicker deposits of ink than the rest of the stroke, these deposits of ink take longer to completely oxidize (compared to the rest of the stroke) and, as a result, remain darker much longer than the rest of the stroke. The deposits have some varying characteristics depending on the type of pen that created the stroke.

The deposit at the end of the stroke of the felt pen is a very distinct and round circle that is darker than the ink around this spot. This deposit fills the entire area at the end of the stroke. The gel pen has a small, circular dark spot located near that end of the stroke that does not fill up the entire end area, as the felt pen does, but is located amidst the thinner layers of ink that are much lighter than the dark ink spot. The deposits of the roller ball stroke are more elongated and stretch a ways up the length of the line. These deposits start lighter and gradually become darker as it approaches the end of the stroke (see fig. 10).

The ballpoint pen is the only type of pen found that does not create a pen stroke with thicker deposits of ink at the end of the stroke. The end of the stroke is completely uniform with the ink of the rest of the stroke. However, the beginning of the ballpoint pen stroke is uniquely lighter than the rest of the stroke. Thus the lighter start and uniform end of a ballpoint stroke is, in itself, a characteristic that discriminates it from the rest of the pen strokes. Since all of the other pen types seems to apply ink strokes which begin uniformly and end with very distinctive thick deposits of ink that require a longer time to oxidize, it could be assumed that the stroke without these unmistakable ink spots is a stroke resulting from a ballpoint pen (see fig. 10).

Because of these deposits of ink at the end of the strokes, each particular character, such as a number or a letter, has a certain, unique pen stroke that can be revealed when exposed to atomic oxygen (see figs. 11 and 12). That is to say, each character has general areas on the pen stroke in which ink is normally deposited. When one character is altered into another one, there is an entirely new stroke being applied on the original marking. The alteration stroke will, like all other strokes, have a thicker deposit at the end of the stroke, which causes there to be a thicker deposit on the altered character where it does exist on a genuine character. When the altered character is atomic oxygen exposed, the resulting thicker deposit of ink from the alteration stroke makes a darker spot on the character that would not be there if the character were genuine, and, as a result, it is apparent that the stroke has been altered.

Some numbers can be changed to create a very realistic and believable alternative number. The number “1” can be altered to form the numbers “4”, “7”, and “9” and the number “3” to the number “8” (see fig. 13). These numbers may be altered in such a manner that their appearance is indistinguishable with a genuine number. The thicker deposits of ink from the alteration stroke are invisible and completely uniform with the ink in the rest of the character. However, similar to the rest of the ink accumulations that are exposed to atomic oxygen, these areas become dark spots that appear at the end of the stroke, creating new spots on the altered character. Thus, the altered characters have ink spots appear on areas of the character that ink spots would not appear on for an unaltered character, creating an obvious telltale indication of alteration. As can be seen in figure 13, the black circles on the exposed drawings exaggerate the general location of where the ink accumulations usually occur for both altered and genuine numbers. For the altered numbers “4,” “7,” and “9,” the ink deposits appear in all the locations that they do with the genuine number, but they also appear in more places than is expected. For the altered number “8,” the ink accumulation areas appear only in areas that the genuine number does not have ink accumulation. The ink samples tested all were visually uniform throughout the character and were without any distinguishing features prior to atomic oxygen exposure, but, as can be seen in figure 14, atomic oxygen successfully uncovered many areas of ink accumulation. It is very apparent that some of the numbers had been altered due to the location of the dark ink spots, and all of the altered numbers had ink accumulate in areas that is unnatural to the original unaltered number.

Effects of careful slow alteration.

An additional finding that was consistently observed for atomic oxygen exposed altered characters was that the altered strokes were always much darker than the original strokes (even made with the same pen). As can be seen in figure 14, the part of the “8” created by the alteration of the number “3” is much darker than the original “3”. This is thought to be due to the fact that careful and accurate alteration requires one to write very slowly to accurately align the strokes with the original character. The slow writing apparently deposits much more ink that the casual quick strokes of the original unaltered character. The alteration therefore has more ink to be oxidized than the original stroke, and, as a result, would remain darker while the original stroke became much lighter.

Effects of paper texture.

One factor that drastically affected how the ink would appear after atomic oxygen exposure is the paper that the ink was written upon. The texture of the paper influences the deposition of ink on the paper and the degree to which it can dry and layer upon itself if altered. The use of atomic oxygen to identify alteration characteristics of the ink or pen stroke relies on the way that the ink layers. On the smooth paper, it was expected that the ink would layer the most ideally, since the crevices that exist on the paper that the ink can flow into were at a minimum. On the porous paper, ink could flow into the crevices, so the layering of the ink would not be as profound. However, unexpectedly, atomic oxygen uncovered the different characteristics of the ink more effectively on the porous paper compared to the smooth paper. The ballpoint and gel pen had difficulties writing a consistent, uniform stroke of ink on the smooth paper. Since the ink strokes were so irregular, it would be impossible for the ink to layer as expected and, therefore, react with atomic oxygen as expected. The felt and roller ball ink changed little after exposure to an atomic oxygen fluence that all the other samples were exposed to (1.41×10^{15} atoms/cm^2), even though the rest of the samples had drastically faded. These inks needed to be exposed to a much larger fluence to be affected. None of the ink on the porous paper appeared as inconsistent as the ballpoint and gel pen on smooth paper. Most of the samples on porous surface paper showed layering and accumulations of ink where these thicknesses were expected to appear. Therefore, as an unpredicted result, the ideal type of paper for atomic oxygen to reveal discriminating results seems to be on porous paper such as normal check stock.

Summary

Atomic oxygen has been found to be a successful tool that can be used to identify certain characteristics of ink and pen strokes. The appearance of ink strokes after atomic oxygen exposure can be applied to determine document alteration. It is possible to identify some document alterations because of the different oxidation rates of different inks, delayed alteration of an ink stroke may add to thickness at the crossing of the two inks, and there may be thicker deposits of ink at the end of a stroke. Different types of ink may look identical before they are exposed, but atomic oxygen can reveal discriminating qualities for various strokes that suggest that a document could have been altered. Atomic oxygen may be able to remove different inks from documents at different rates, and may uncover thicker areas of ink, such as deposits at the end of the stroke and at delayed line crossings. Also, because alteration strokes are generally written slower than the original stroke, the altered stroke is much thicker. This thicker alteration stroke takes longer to oxidize than the thinner original stroke, thus the alteration stroke appears much darker than the original stroke. The exact appearance of an atomic oxygen exposed ink stroke is also dependent on many factors, such as the type of pen, type of ink, color of ink, and paper texture. Some types of ink will react more ideally with atomic oxygen when written on certain papers or with a certain brand. There are some circumstances when atomic oxygen does not react as ideally with inks. In some situations, atomic oxygen has revealed characteristics of the ink that contradict the idea of the way that atomic oxygen is expected to react with inks. Although identification of character alteration using atomic oxygen is not a certainty, the technique can usually be used to provide strong suspicion of document alteration.

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TABLE I.—PENS USED FOR ATOMIC OXYGEN EXPOSURE TESTING

Type	Brand	Color
Ballpoint Pen	Bic	black
Ballpoint Pen	Bic	blue
Gel Pen	PenTech	black
Gel Pen	PenTech	blue
Roller Ball Pen	Sanford	black
Roller Ball Pen	Sanford	blue
Felt Tip Pen	Paper Mate	black
Felt Tip Pen	Paper Mate	blue
Felt Tip Pen	EF	black
Felt Tip Pen	Unlabeled (fine point)	black
Felt Tip Pen	Expresso (Extra Fine)	black
Felt Tip Pen	Expresso (Medium)	black
Sharpie Permanent Marker	Sanford (Ultra Fine Point)	black

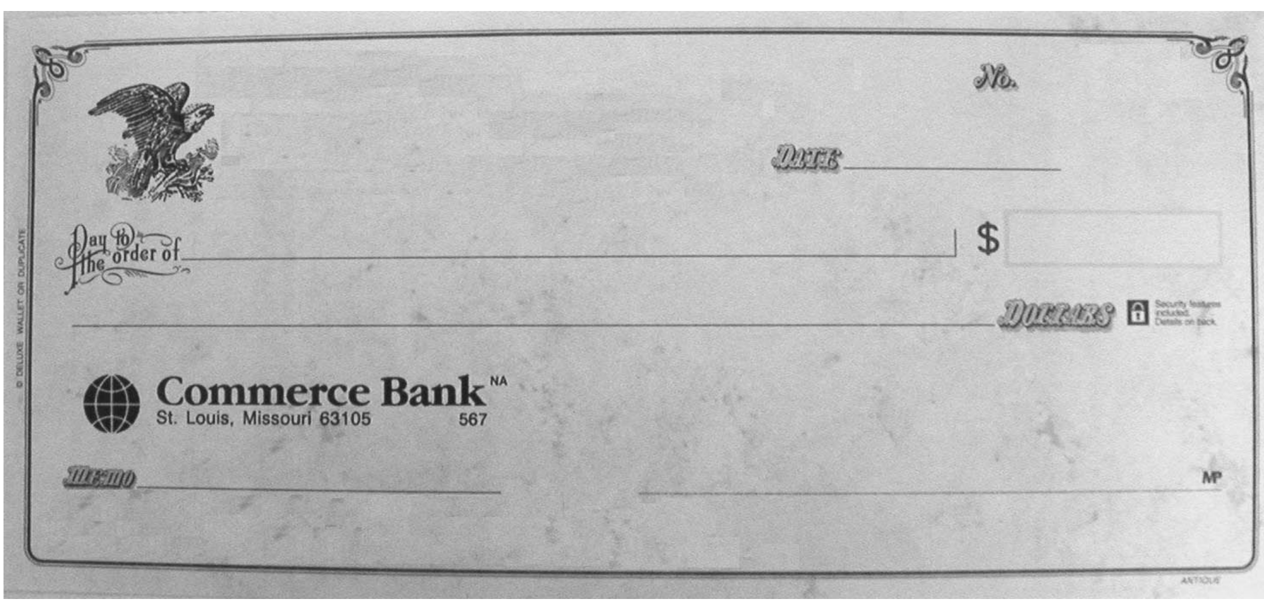
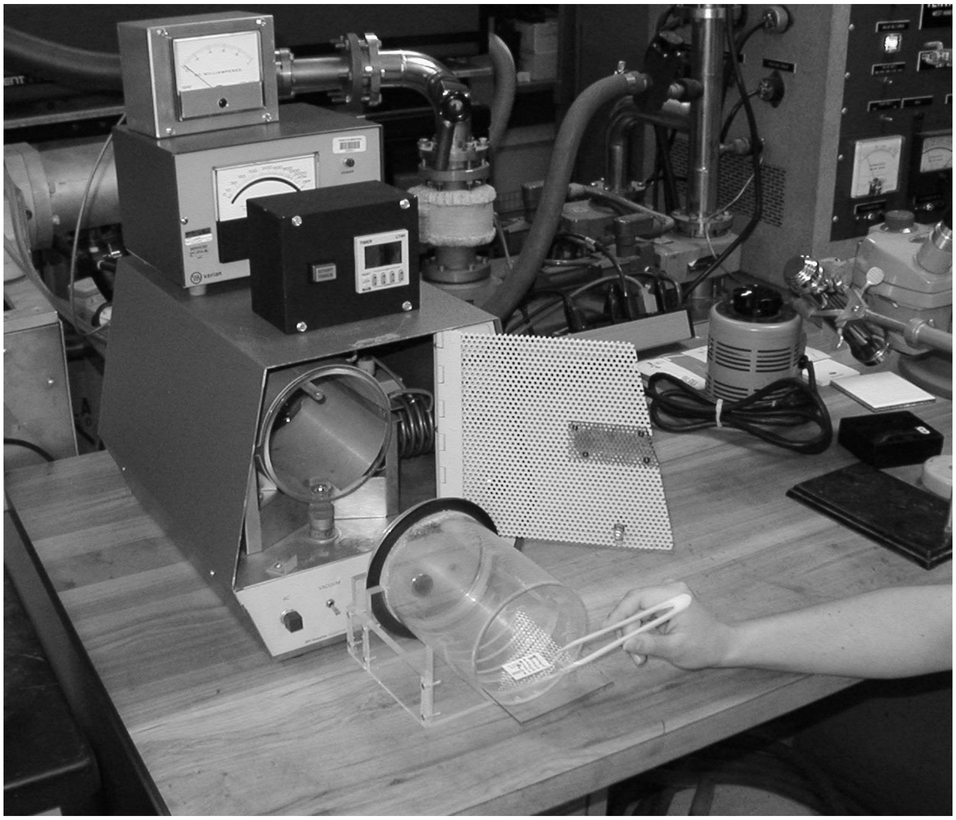


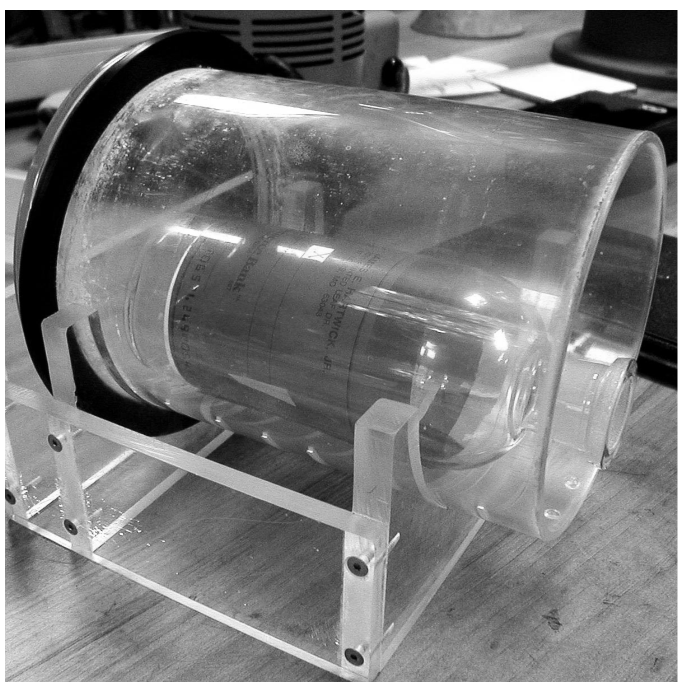
Figure 1.—Photograph of check stock used for atomic oxygen testing.



(a). A sample of check stock with ink marks on it being placed on a glass tray for insertion into the plasma asher.

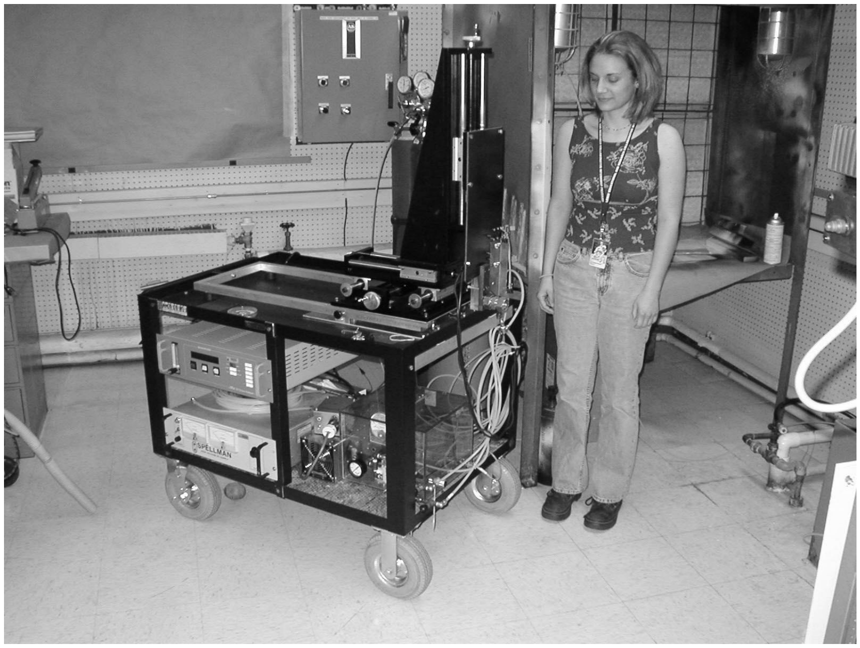
(b). Plasma asher during operation.
Figure 2.—SPI Plasma Prep II Plasma Asher.

(a). Kapton protects the document from atomic oxygen exposure except for a small window cut in the questioned area.

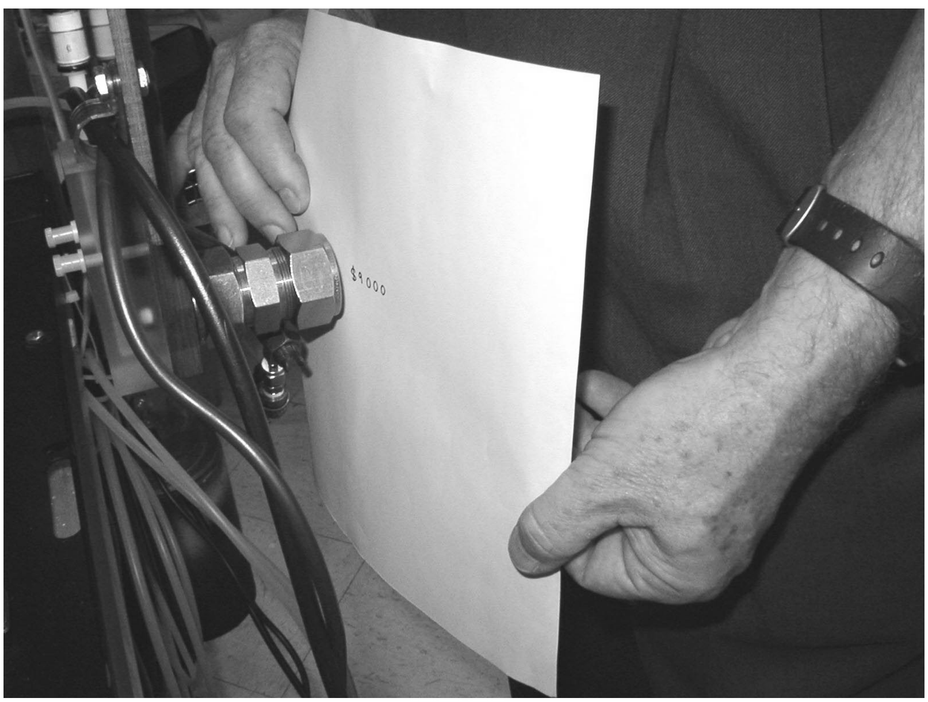


(b). The glass bottle is placed in the glass cylinder of the asher and then inserted into the machine.

Figure 3.— Protective masking of a small document in order to control the area exposed to atomic oxygen.

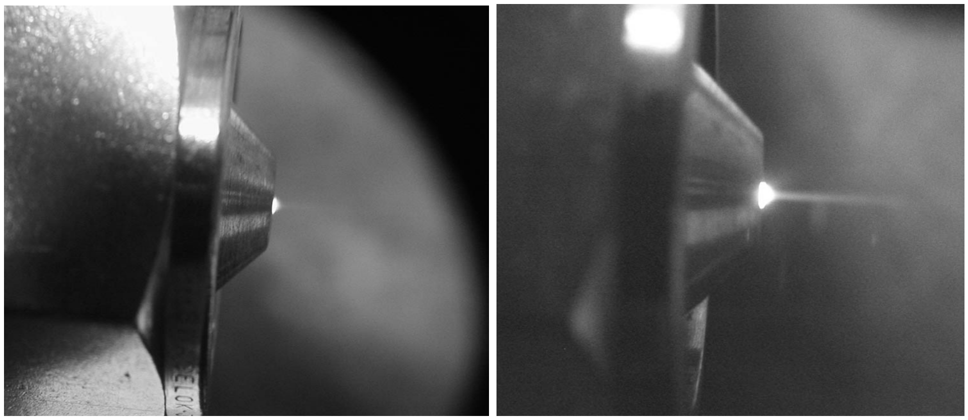


(a). Overall photo of the atmospheric atomic oxygen beam system.



(b). Sample of paper being exposed to the atmospheric atomic oxygen beam.

Figure 4.—Atmospheric atomic oxygen beam.



In room light | Time exposure in the dark

(c). A close photograph of the atmospheric atomic oxygen beam and nozzle.

Figure 4.—Concluded.

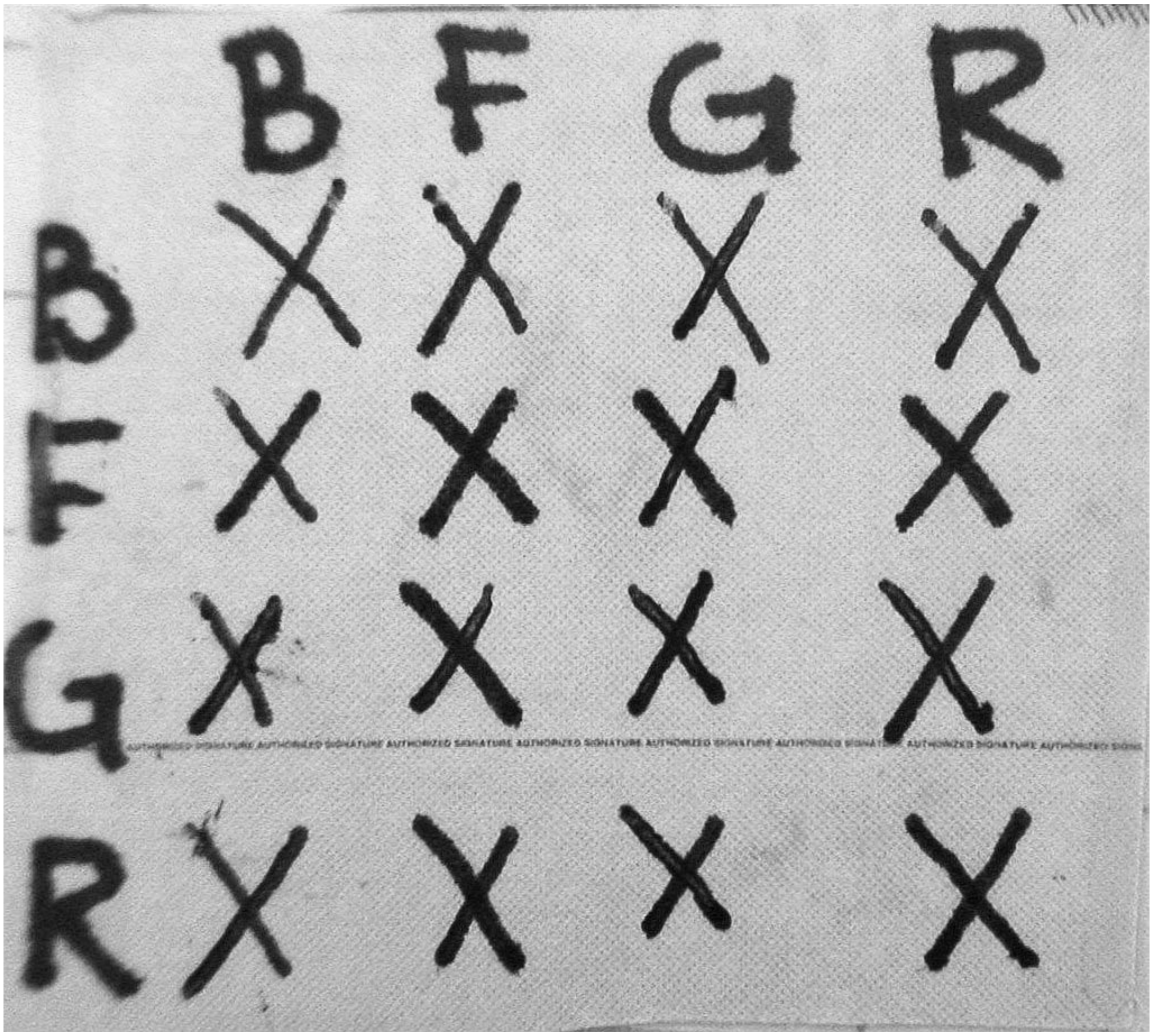
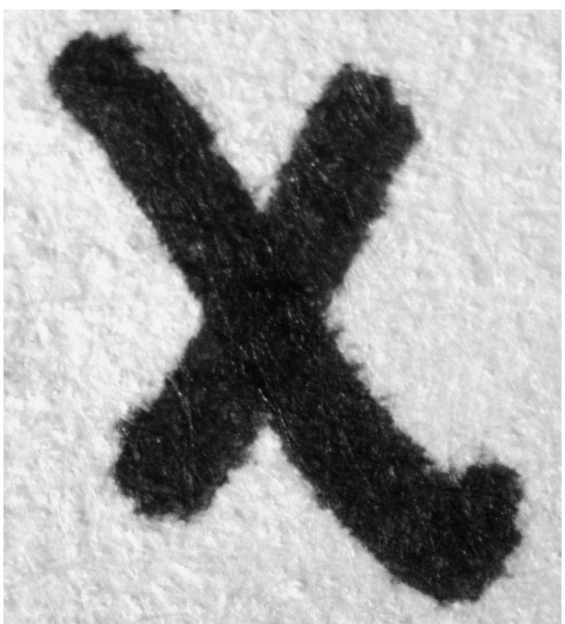


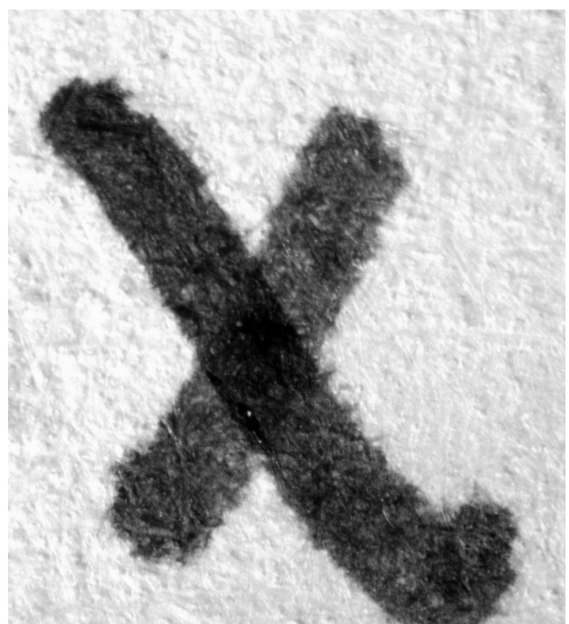
Figure 5.—Pen mark "X" matrix on sample of check stock prior to atomic oxygen exposure.

Felt Tip Pen Ink before exposure (no delay)

Felt Tip Pen Ink after exposure (no delay)



Felt Tip Pen Ink before exposure (with delay)



Felt Tip Pen Ink after exposure (with delay)

Figure 6.—Ink thickness differences with and without delay in making an "X" prior to and after atomic oxygen exposure.

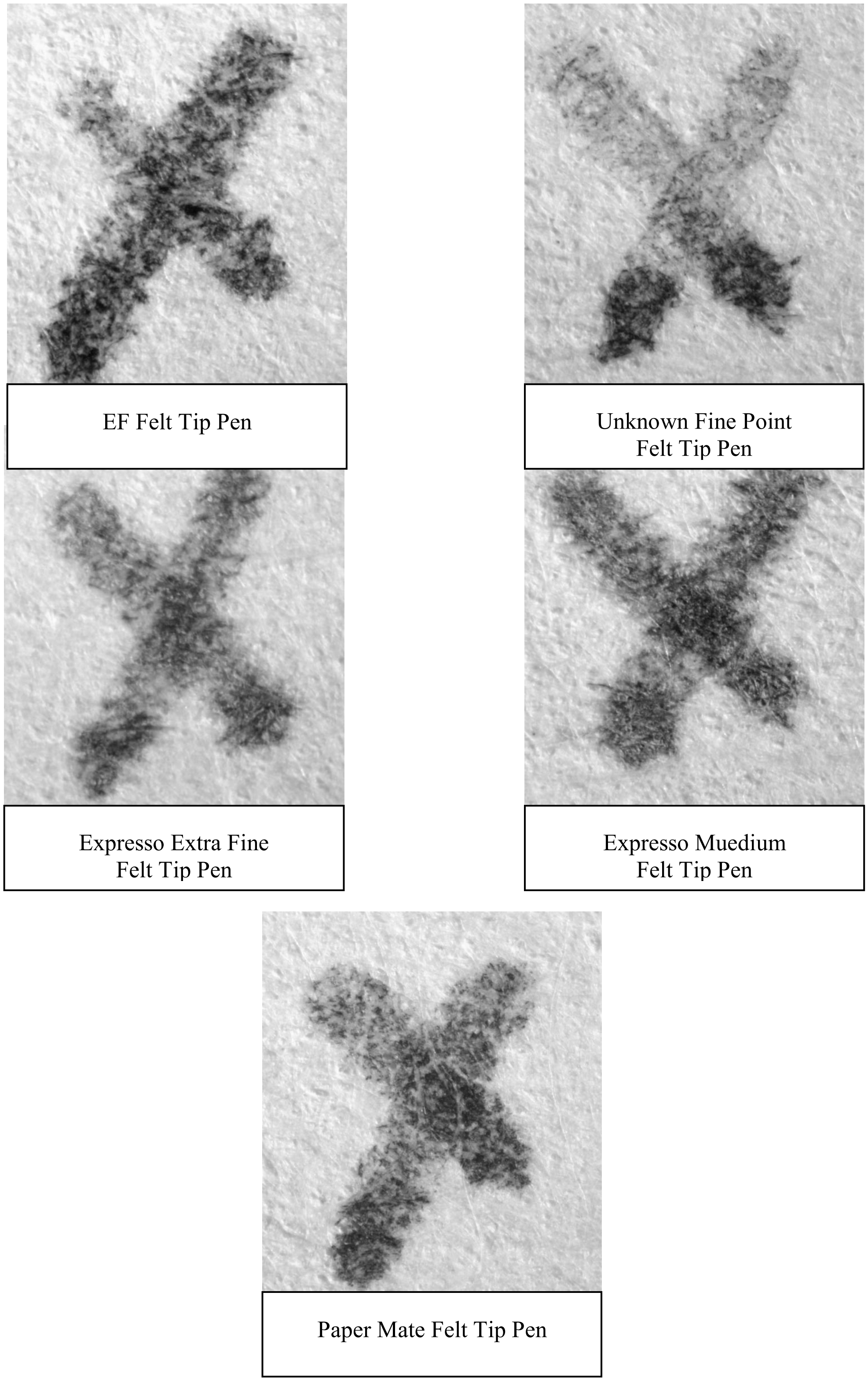
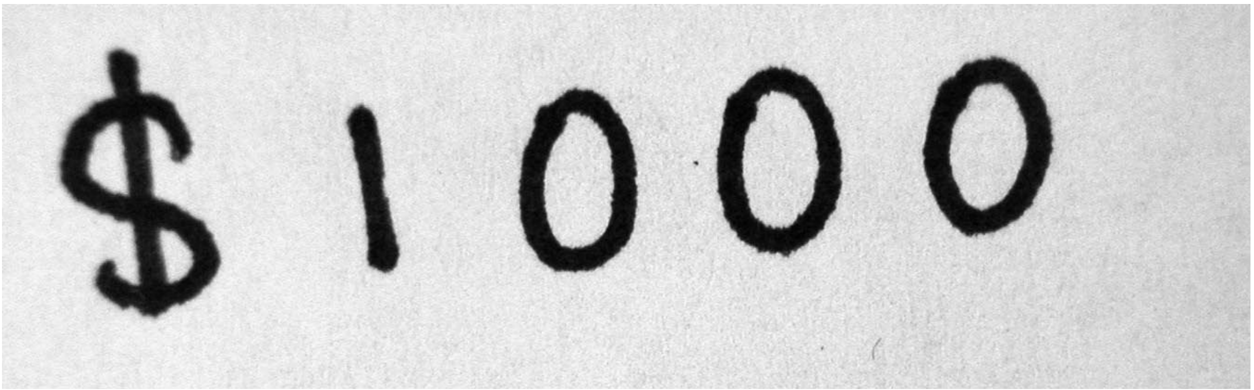
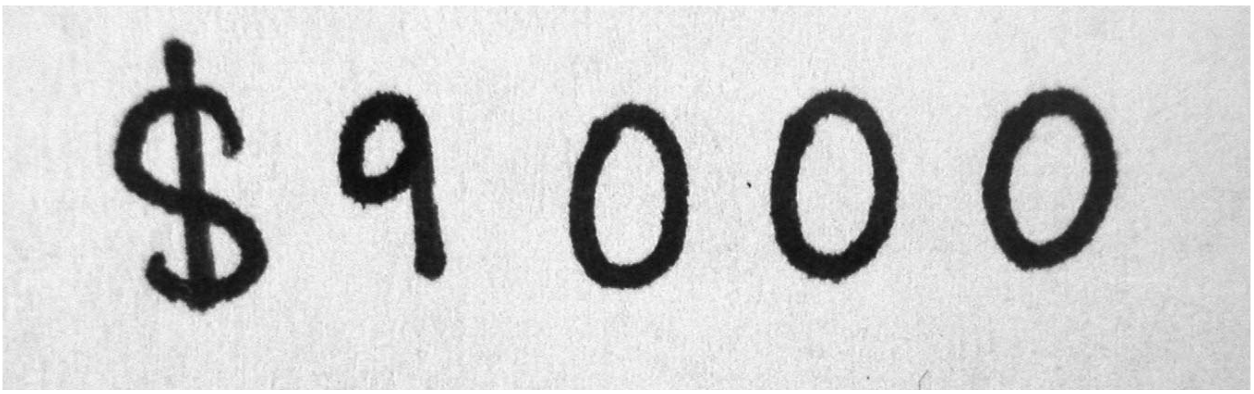


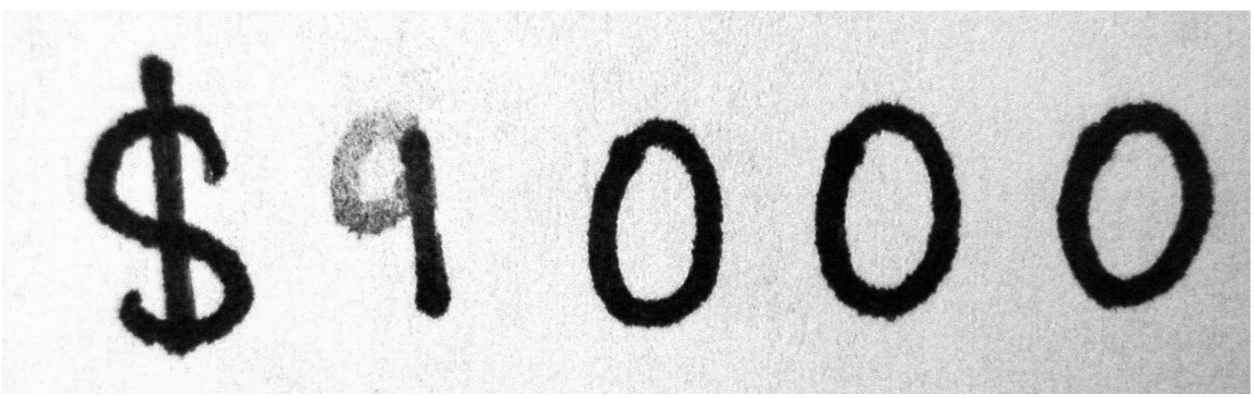
Figure 7.—Different inks from felt tip pens after atomic oxygen exposure.



(a). Unaltered permanent marker ink prior to atomic oxygen exposure

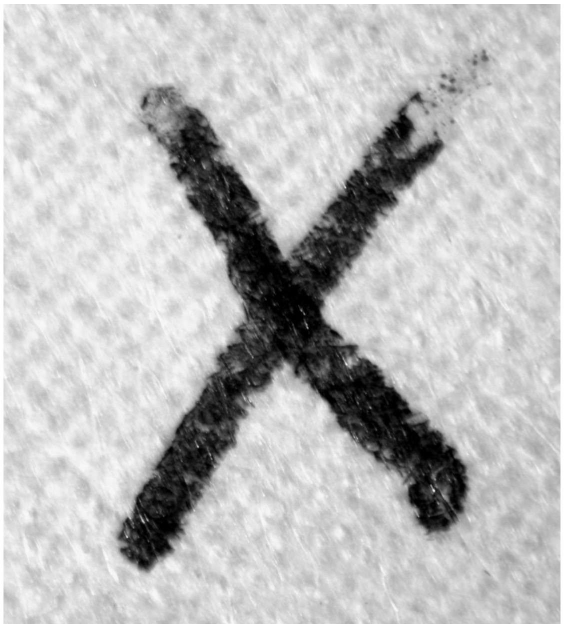


(b). Altered permanent marker ink with felt tip pen (atomic oxygen removable ink) prior to exposure to atomic oxygen



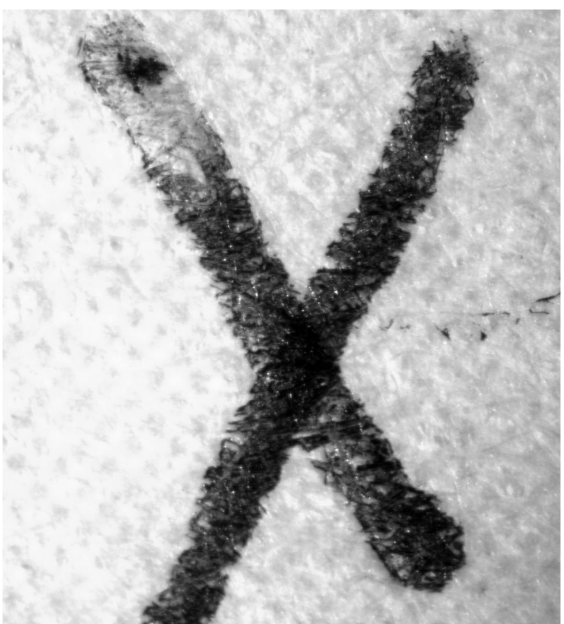
(c). Altered permanent marker number after atomic oxygen exposure; atomic oxygen removable ink has been significantly oxidized.

Figure 8.—Demonstration of the inks' different oxidation rates.

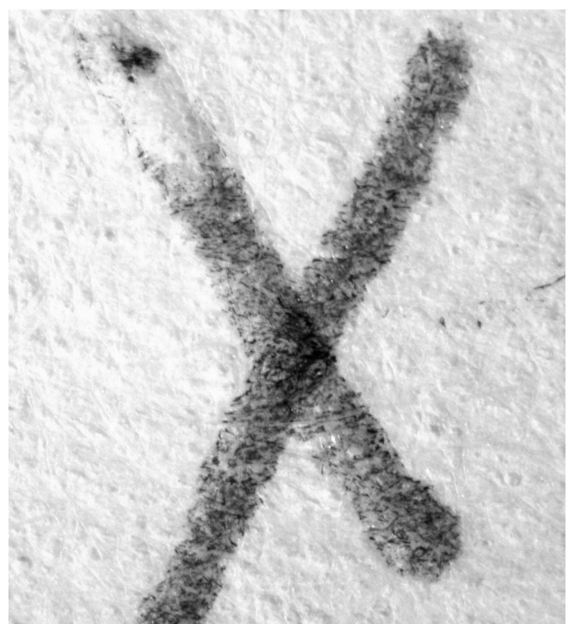


Ballpoint Pen Ink before exposure (no delay)

Ballpoint Pen Ink after exposure (no delay)

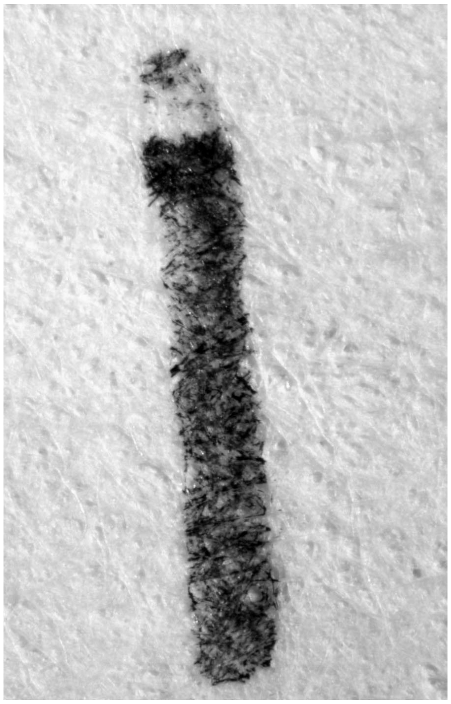


Ballpoint Pen Ink before exposure (with delay)

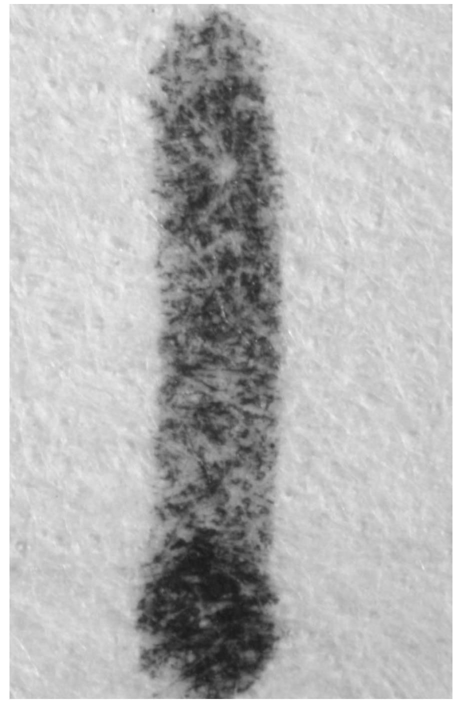


Ballpoint Pen Ink after exposure (with delay)

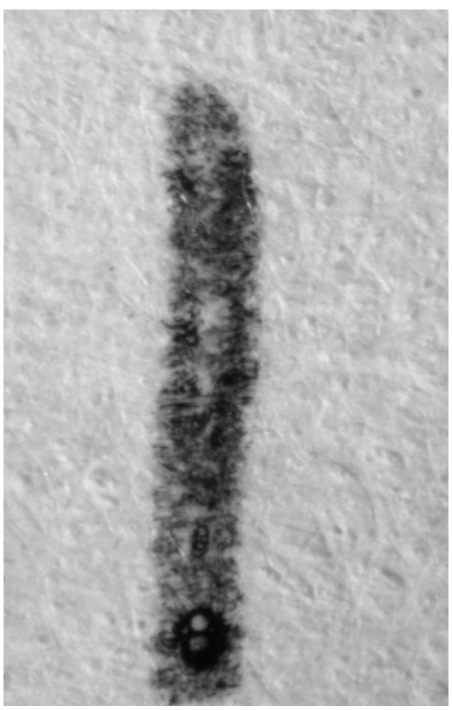
Figure 9.—Ink thickness with and without delay in making an "X" prior to and after atomic oxygen exposure in which exposing the top ink sample generated results opposite to what would be expected.



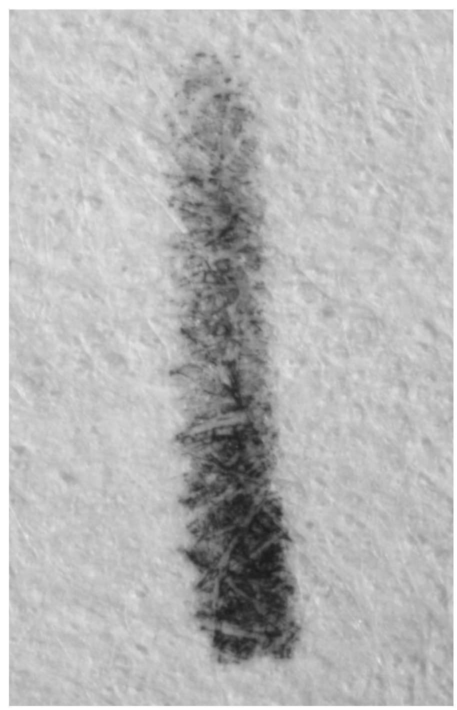
(a). Ballpoint pen.



(b). Felt tip pen.



(c). Gel ink pen.



(d). Roller ball pen.

Figure 10.—Various types of black pen strokes after atomic oxygen exposure.

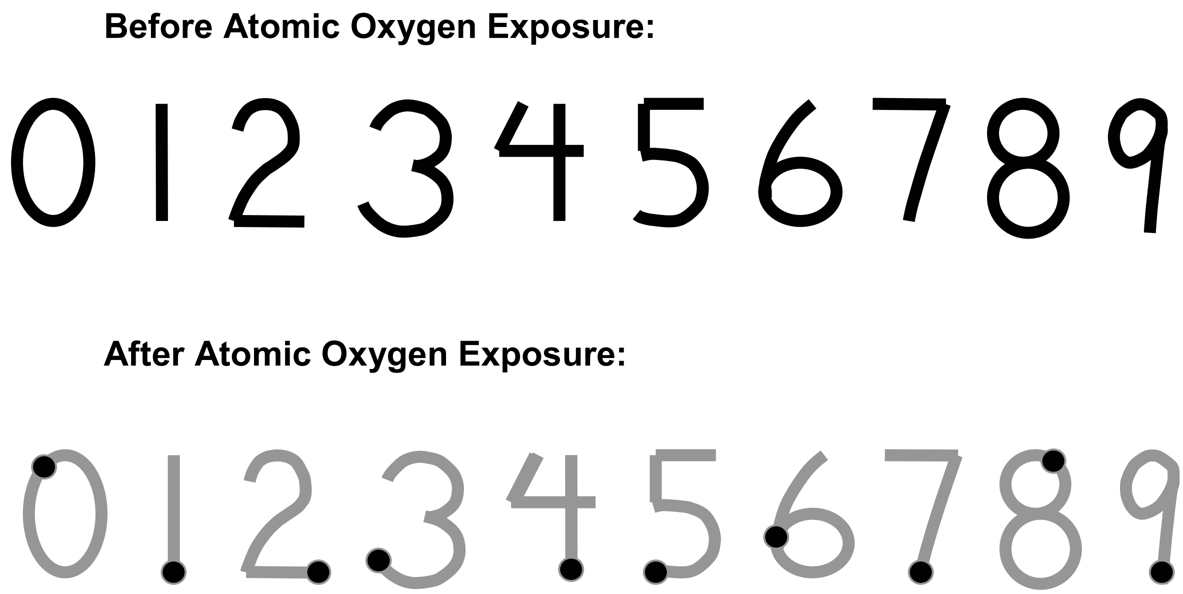


Figure 11.—Diagram of a visualization of ink accumulation at the end of pen strokes. The black dots of the exposed drawings exaggerate the location of where the ink accumulation occurs.

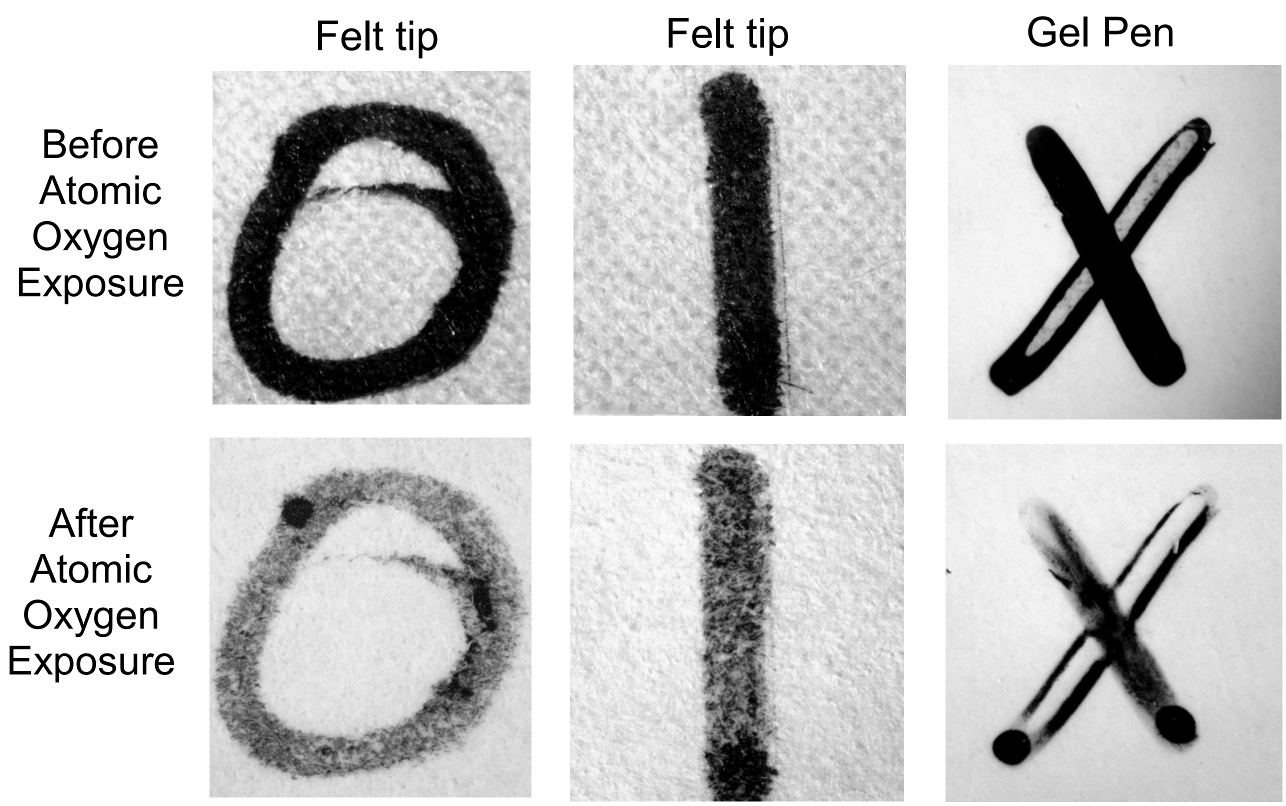


Figure 12.—Pen strokes for unaltered numbers before and after atomic oxygen exposure.

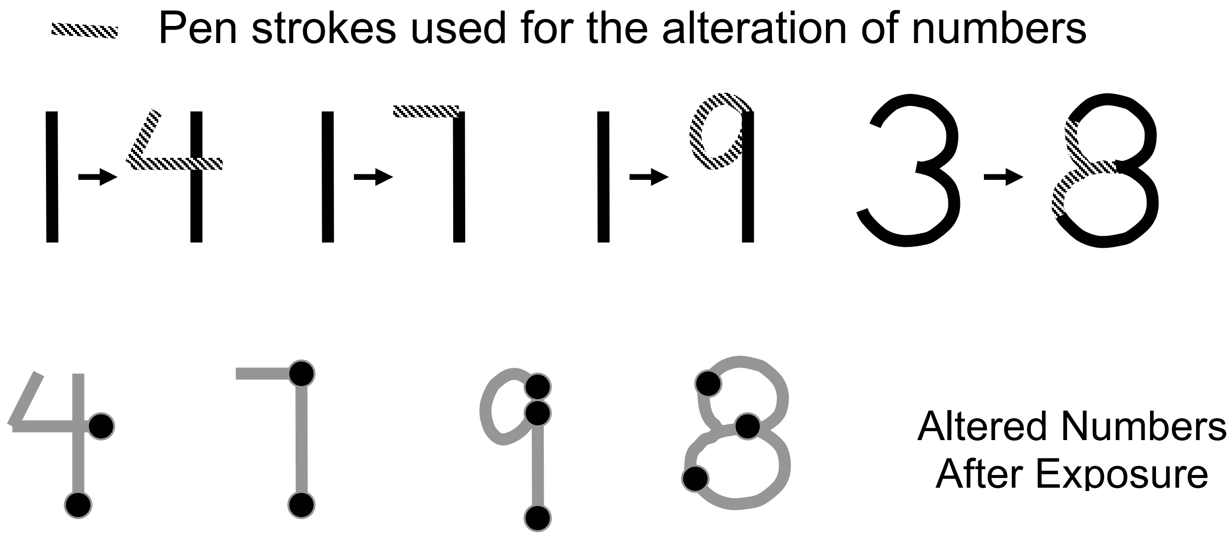


Figure 13.—Ink accumulation at end of pen strokes for altered numbers.

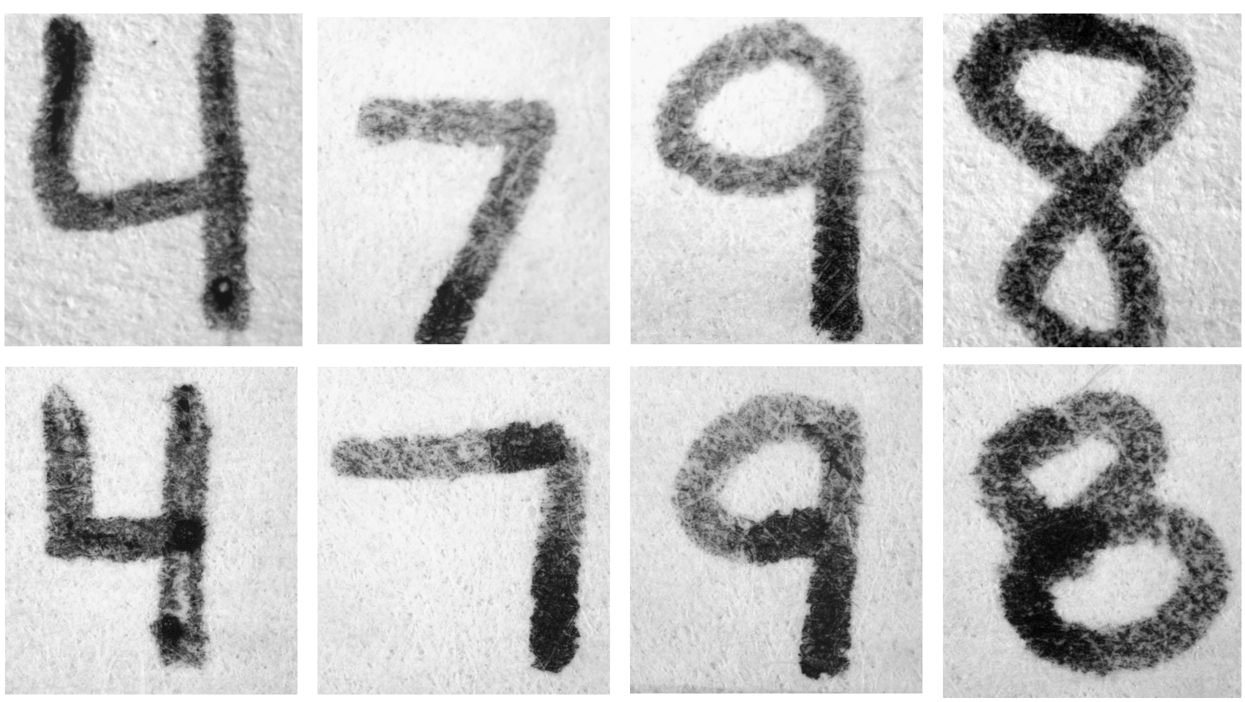


Figure 14.—Altered and unaltered numbers after atomic oxygen exposure. The numbers in the first row are unaltered numbers. The numbers in the second row are altered numbers from original numbers 1,1,1 and 3 respectively from left to right.

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13. ABSTRACT *(Maximum 200 words)*

Atomic oxygen, which normally is found only the near Earth space environment, causes oxidation and erosion of polymers on spacecraft. The development of technology to prevent this degradation has required NASA to develop ground laboratory facilities that generate atomic oxygen. Atomic oxygen has also been found to be able to oxidize most types of ink from a variety of types of pens. The use of atomic oxygen to identify alteration of documents has been investigated and is reported. Results of testing indicates that for many types of ink, pen, and paper, identification of document alteration of pen and ink numbers and evidence of alteration can be made visible by exposing the questionable writing to atomic oxygen. Atomic oxygen provides discrimination because different inks may oxidize at different rates, the amount of time between delayed alteration may add to ink thickness at crossings, and the end of pen strokes tend to have much thicker ink deposits than the rest of the character. Examples and techniques of using atomic oxygen to identify document alteration indicate that the technology can, in many but not all cases, provide discrimination between original and altered documents.

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