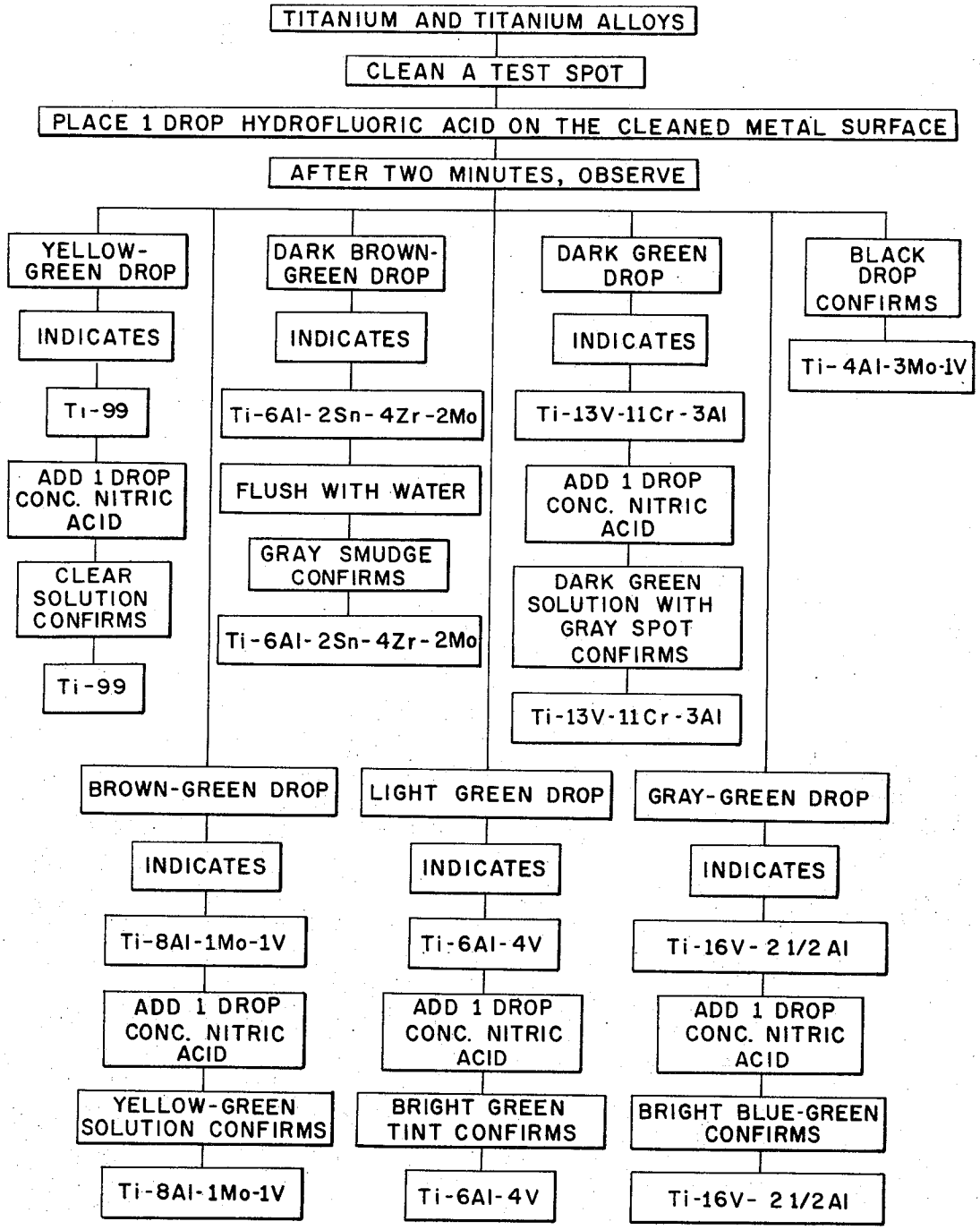
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NONDESTRUCTIVE SPOT TEST METHOD FOR TITANIUM AND TITANIUM ALLOYS
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NONDESTRUCTIVE SPOT TEST METHOD FOR TITANIUM AND TITANIUM ALLOYS

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ABSTRACT OF THE DISCLOSURE

A method for spot test identification of titanium and various titanium alloys commonly used in aerospace applications. The spot test identification involves color codes obtained when a drop of concentrated hydrofluoric acid and a drop of concentrated nitric acid are placed on the surface to be tested.

ORIGIN OF THE INVENTION

This invention was made by an employee of the National Aeronautics and Space Administration and may be manufactured and used by or for the Government of the United States of America for governmental purposes without the payment of any royalties thereon or therefor.

The invention relates to an improved spot test procedure to identify titanium and various titanium alloys commonly employed in aerospace applications. More particularly, the invention is related to a simple chemical spot test color developing when a titanium or titanium alloy is treated with a drop of concentrated hydrofluoric acid followed by a drop of concentrated nitric acid.

The invention is particularly suited for use by shop and laboratory personnel having no special training in the field of chemistry with conclusions relative to identification being derived from unique reactions or colors being produced by the acid reactions.

To assure quality fabrication of hardware and maximum safety to personnel, it is necessary to identify or verify the various metals and alloys involved. That is, if two or more components are to be intelligently joined together by modern welding techniques, their positive identities are essential. Present facilities for the identification of various metals and alloys frequently require time consuming procedures of chemical or spectrographic analyses.

It is therefore an object of the present invention to provide a new and novel nondestructive identification process for titanium and titanium alloys.

Another object of the present invention is a simple chemical test identification process for titanium and titanium alloys that can be performed by shop or laboratory personnel that have no special training in chemistry.

Another object of the present invention is a color spot test for identifying titanium and titanium alloys.

Another object of the present invention is a process for identifying alloys in titanium alloys.

A further object of the present invention is a semi-quantitative technique for various alloys contained in titanium alloys.

The foregoing and other objects are attained by taking a metal known to be titanium or a titanium alloy, cleaning a small surface area thereon by use of a file, abrasive cloth

or suitable solvent, placing one drop of concentrated hydrofluoric acid on the clean surface, allowing it to react for two minutes and then observing the color of the drop. The color of the drop identifies the sample as titanium or one of its alloys according to the colors obtained. A standard chart containing the color code may be referred to for positive identification or the test may be conducted simultaneously on a sample of known composition. Further identification of the particular alloy can be confirmed by adding one drop of concentrated nitric acid to the colored drop and observing the color change.

A more complete appreciation of the present invention and many of the attendant advantages thereof will be readily appreciated as the same becomes better understood by reference to the following detailed description when considered in connection with the accompanying drawing wherein:

The single figure of the drawing is a flow sheet representing the process for differentiating and identifying titanium and various titanium alloys.

Referring to the figure, it is assumed that it is known that the particular sample to be tested and identified is known to be in the titanium family, that is, titanium-99 or a specific titanium alloy. A small test area on the specimen is then cleaned to remove any dirt, paint, grease, oxide or added metallic coating prior to performing the test. This cleaning process may involve the use of conventional cleaning solvents, files, abrasives or the like. After proper cleaning the analyst should follow the chart in detail as set forth in the figure. Initially, one drop of concentrated hydrofluoric acid (48%) is placed on the cleaned metal surface, allowed to react for two minutes and the color of the drop observed. For color comparison, the analyst may compare the drop with a standardized color chart prepared for that purpose from observations taken from known titanium and titanium alloys or he may simultaneously test a sample of known material along with the unknown and make the comparison therewith. In any event, the color obtained is clear and distinct.

A yellow-green color formed by the hydrofluoric acid drop indicates Ti-99. This is confirmed by adding one drop of concentrated nitric acid to the previous drop and if the yellow-green color disappears and the drop becomes colorless, the unknown is, in fact, Ti-99.

A light brown-green color formed by the hydrofluoric acid drop indicates that the unknown is the alloy Ti-8Al-1Mo-1V. This is confirmed by adding one drop of concentrated nitric acid to the previous drop and if the unknown is, in fact, Ti-8Al-1Mo-1V, the two acids will form a yellow-green solution.

A dark brown-green color formed by the hydrofluoric acid drop indicates that the unknown is the alloy Ti-6Al-2Sn-4Zr-2Mo. This is confirmed by washing away the acid drop with distilled water and if, upon drying, a gray smudge remains the unknown is, in fact, Ti-6Al-2Sn-4Zr-2Mo.

A light green color formed by hydrofluoric acid drop indicates that the unknown is Ti-6Al-4V. This is confirmed by adding one drop of concentrated nitric acid to the previous drop and if, in fact, the unknown is Ti-6Al-4V, the color of the combined acids will change to a bright green tint.

A dark green color formed by the hydrofluoric acid drop indicates that the unknown is Ti-13V-11Cr-3Al. This is confirmed by adding one drop of concentrated

nitric acid to the previous drop and if the unknown is Ti-13V-11Cr-3Al the color of the combined drops will remain dark green, but clear, revealing a gray spot on the surface of the metal beneath the solution.

A gray-green color formed by the hydrofluoric acid drop indicates that the unknown is Ti-16V-2½Al. This is confirmed by adding one drop of concentrated nitric acid to the previous drop and if the gray-green color changes to a bright blue-green color for the combined drops the unknown is, in fact, Ti-16V-2½Al.

A black color formed by the hydrofluoric acid drop indicates that the unknown is Ti-4Al-3Mo-1V. No confirmation test is necessary since the black drop is indicative of this alloy only.

The above described tests are normally sufficient to positively identify the titanium alloys; however, additional color spot tests have been developed to identify specific metals alloyed with titanium. For example, a test for aluminum alloyed with titanium would be as follows: (a) place one drop of concentrated hydrofluoric acid on the clean metal surface (cleaned as described hereinbefore) of the unknown. After one minute add three drops of concentrated nitric acid and one minute thereafter, add six drops of distilled water and transfer the resulting solution to a small test tube; (b) add 0.5 milliliter of 6 M sodium hydroxide and stir with a glass rod. After thoroughly mixing add an additional 0.5 ml. of the 6 M sodium hydroxide and (c) place the test tube in a boiling water bath for three minutes. Remove the tube from the bath and centrifuge for approximately five minutes at about 1725 r.p.m., (d) transfer three drops of the supernatant liquid to a spot plate depression, acidify with sixteen to twenty drops of 1 M acetic acid, add two drops of alizarin reagent and stir with a glass rod. After five minutes, add two drops of 1 M hydrochloric acid, stir and observe after five to ten minutes. A red-to-orange color indicates the presence of aluminum while, if no aluminum is present to react with the acidified solution of alizarin, the solution turns yellow. The alizarin solution is prepared by dissolving 0.06 gram of Alizarin Red S (Allied Chemical Corporation, National Aniline Division, 40 Rector St., New York 6, N.Y.) in 60 ml. of distilled water. If the red-to-orange color is formed, a semi-quantitative determination of the aluminum can be obtained by comparing the intensity of the color obtained with a color chart indicating the color intensity for titanium alloys having known aluminum contents.

To test for molybdenum alloyed with titanium, two approaches are available. First, one drop of concentrated hydrofluoric acid is placed on a cleaned surface of the metal to be tested and allowed to react for one minute. This is followed by adding a drop of concentrated nitric acid to the spot, stir with a glass rod and after one minute add a drop of distilled water. Two drops of the combined liquids are then taken up by means of a medicine dropper and transferred to a white porcelain spot plate. Two drops of ten percent potassium thiocyanate solution and one drop of a twenty-five percent sodium thiosulfate solution are added to the acid solution in the spot plate depression and the various drops stirred together. If molybdenum is present, a pink-to-violet color develops after the brown color that was formed by the addition of the potassium thiocyanate disappears. The intensity of the pink-to-violet color is proportional to the molybdenum content and is readily checked by reference to a standardized color chart. Metals containing one percent and three percent molybdenum, respectively, are easily differentiated by this method.

In a different approach to test for molybdenum, two or three drops of a solution consisting of 25% concentrated nitric acid plus 5 percent concentrated hydrofluoric acid plus 70 percent distilled water, all by volume, are added to the cleaned metal surface and allowed to react for two minutes. One drop of 6 M hydrochloric acid is then placed on a filter paper disc (#2 Whatman qualitative filter paper circles or the equivalent are very convenient for this test) and one drop of the acid test solution is transferred from the metal to the center of the wetted fleck. One drop of ten percent potassium thiocyanate and one drop of twenty-five percent sodium thiosulfate are then added to the center of the fleck. A pink-to-violet color develops in the center of the fleck extending outward forming a pink-to-violet ring around the center fleck if molybdenum is present. Again, the intensity of the color obtained is proportional to the molybdenum content.

To test for tin a titanium alloy, a fluoroboric acid solution is formed by dissolving 28 grams of boric acid in 60 ml. of 48 percent hydrofluoric acid. This is stored in a polyethylene bottle until ready for use and ten drops of this acid solution placed on the cleaned surface of the metal to be tested. After allowing five minutes for reaction time, gently flush the spot with distilled water, washing off the liquid acid solution. The spot is then dried in a gentle stream of air, leaving a gray-to-black powdery precipitate on the surface of the metal if tin is present. To confirm, add ten drops of concentrated hydrochloric acid to the spot on the metal, stir with a glass rod, and after allowing five minutes reaction time transfer five drops of the acid solution to a small test tube. Add five drops of distilled water. At this point, the test solution may be centrifuged for 3 to 5 minutes at 1725 r.p.m. if desirable, and the clear liquid solution transferred to a clean test tube by pouring, discarding the solid residue. However, tin can be confirmed with or without centrifuging to eliminate the solid particles as above mentioned. Next, add three drops of a saturated solution of mercuric chloride (saturated in distilled water).

A white-to-gray precipitate confirms the presence of tin, the quantity of which is proportional to the content of tin present in the metal.

An alternate method for determining the presence of tin in titanium alloys is the following: heat the sample metal to approximately 300° F. and place ten drops of concentrated hydrochloric acid on the cleaned surface of the metal. After three-to-five minutes transfer five drops of the acid solution to a small test tube. (If the concentrated hydrochloric acid evaporates too rapidly, 5 to 10 drops of additional concentrated hydrochloric acid may be added and immediately take up 5 drops with a medicine dropper and place them in the test tube.) Next, add 5 drops of distilled water, stir by moving the test tube in a circular motion and add 3 drops of a saturated solution of mercuric chloride (before mentioned). A milky white-to-gray precipitate confirms the present of tin. The tin content of the metal is proportional to the precipitate formed. A semi-quantitative determination of tin can be made using this method if titanium alloys containing known amounts of tin are tested simultaneously for comparison purposes.

To test for vanadium alloyed in titanium and titanium alloys the metal surface is cleaned as described hereinbefore and one drop of concentrated hydrofluoric acid placed on the clean surface. After one minute, three drops of 1:1 nitric acid are added and after one minute, the solution is stirred with a glass rod. If a brown color appears and remains at this point, an additional two drops of 1:1 nitric acid are stirred in. A high vanadium content is indicated by a bright blue-green color in the acid solution. Two drops of this blue-green solution are transferred to a spot plate and two drops of phosphoric acid and one drop of α,α' dipyridyl reagent (prepared by dissolving 1.2 grams of α,α' dipyridyl in 60 ml. of 0.1 M HCl), stirred therein. This is followed by four drops of concentrated ammonium hydroxide, stirred in, and two drops of concentrated phosphoric acid, stirred in. A pink-to-red color, depending upon the vanadium content identified the presence of vanadium. A semi-quantitative determination is readily made by comparison with standardized color

charts. Commercially pure titanium will produce a white color at this point.

To test for zirconium alloyed in titanium and titanium alloys the metal surface is cleaned as described hereinbefore, eight drops of concentrated hydrochloric acid added to the cleaned metal surface which has been heated to approximately 300° F. on a hot plate, open flame, etc. After allowing the hydrochloric acid to react on the metal for one minute, remove the metal from the heat source and cool to room temperature. Three drops of 2 M sodium hydroxide are placed on a Whatman #3 extra thick qualitative filter paper disk and one drop of the acid solution is taken off of the metal surface and added to the center of the wetted surface of the filter paper. This is followed by one drop of alizarin solution (0.1%) (formed by dissolving 0.06 gram of Alizarin Red S in 60 ml. of distilled water described hereinbefore) added to the center of the fleck and observed for five minutes. A red precipitate in the center of the fleck that is stable (does not dissolve or change color when one or two drops of 1 M HCl is added to the center of the fleck) confirms the presence of zirconium. Again, the intensity of the red color and the quantity of the precipitate is indicative of the quantity of zirconium present.

The advantages of the present invention and its ease of adaptation for use by shop and laboratory personnel having no special training in chemistry are now believed apparent. The conclusions relative to identification are derived from unique reactions or the colors produced by the addition of the various reagents and the colors obtained may be readily compared with standardized color charts prepared from known metal tests. The various tests described herein are nondestructive in that they are conducted on the metal surface and the material affected is equivalent to the stroke of a smooth file or cleaning with an abrasive cloth. Although the tests are intended primarily as qualitative, semiquantitative conclusions in some instances are readily made by comparison with standardized color charts, simultaneously testing specimens having known chemical compositions and by comparing the relative quantity of precipitates produced or the intensities of the color reactions.

Although the procedures and process steps described in the various tests described herein have been enumerated in detail, the invention is not so limited and there are many variations and modifications of the procedures that will be readily apparent to those skilled in the art in the light of the above teachings. It is therefore to be understood that the invention may be practiced otherwise than as specifically described.

What is claimed as new and desired to be secured by Letters Patent of the United States is:

1. A rapid nondestructive spot test method for differentiating between and identifying titanium and titanium alloys where the test specimen is known to be selected from the group consisting of Ti-99, Ti-8Al-1Mo-1V, Ti-6Al-2Sn-4Zr-2Mo, Ti-6Al-4V, Ti-13V-11Cr-3Al, Ti-16V-2½Al and Ti-4Al-3Mo-1V, comprising:
   - cleaning a test spot on the unknown specimen to remove any dirt, paint, grease, oxide or added metallic coating by use of a cleaning solvent, file, abrasive or the like,
   - placing one drop of concentrated hydrofluoric acid on the cleaned spot,
   - allowing the acid to react for two minutes and observing the color formed by the acid drop.

2. The process of claim 1 wherein the color obtained by the acid drop is a yellow-green color thereby indicating that the unknown specimen is Ti-99 and further including the process step of adding one drop of concentrated nitric acid to the yellow-green drop whereby the disappearance of the yellow-green color leaves a clear acid solution on said unknown and confirms that said unknown is Ti-99.

3. The process of claim 1 wherein the color obtained by the hydrofluoric acid drop is a light brown-green color thereby indicating that the unknown specimen is Ti-8Al-1Mo-1V and further including the process step of adding one drop of concentrated nitric acid to the light brown-green drop and wherein the resulting reaction of the combined acid drops produces a yellow-green solution to confirm that said unknown is Ti-8Al-1Mo-1V.

4. The process of claim 1 wherein the color obtained by the hydrofluoric acid drop is a dark brown-green color thereby indicating that the unknown specimen is Ti-6Al-2Sn-4Zr-2Mo and further including the step of flushing the dark brown-green drop away with distilled water whereby a gray smudge remaining on the unknown specimen confirms that the unknown is Ti-6Al-2Sn-4Zr-2Mo.

5. The process of claim 1 wherein the color obtained by the hydrofluoric acid drop is a light green color thereby indicating that the unknown specimen is Ti-6Al-4V and further including the step of adding a drop of concentrated nitric acid to the light green drop and wherein a resulting bright green tint to the combined acid drops confirms that the unknown is Ti-6Al-4V.

6. The process of claim 1 wherein the color obtained by the hydrofluoric acid drop is a dark green color thereby indicating that the unknown specimen is Ti-13V-11Cr-3Al and further including the step of adding a drop of concentrated nitric acid to the dark green acid drop and wherein a resulting clear dark green solution with a visible grap spot formed on the surface of the metal confirms that the unknown is Ti-13V-11Cr-3Al.

7. The process of claim 1 wherein the color obtained by the hydrofluoric acid drop is a gray-green color thereby indicating that the unknown specimen is Ti-16V-2½Al and further including the step of adding a drop of concentrated nitric acid to the gray-green drop and wherein a bright blue-green color of the combined drops confirms that the unknown is Ti-16V-2½Al.

8. The process of claim 1 wherein the color obtained by the hydrofluoric acid drop is a black color thereby indicating and confirming that the unknown specimen is Ti-4Al-3Mo-1V.

9. A rapid nondestructive spot test method for differentiating between and identifying titanium and titanium alloys where the test material is known to be selected from the group consisting of Ti-99; Ti-8Al-1Mo-1V; Ti-6Al-2Sn-4Zr-2Mo; Ti-NAl-4V; Ti-13V-11Cr-3Al– Ti-16V-2½Al; and Ti-4Al-3Mo-1V, comprising:
   - cleaning a test spot on the unknown specimen to remove any dirt, paint, grease, oxide or added metallic coating thereon by use of a cleaning solvent, file, abrasive or the like,
   - placing one drop of concentrated hydrofluoric acid on the cleaned spot,
   - allowing the acid drop to react for two minutes and observing the color appearing in said drop,
   - comparing the color formed by said acid drop with a color chart having standardized colors thereon coded from tests of a drop of concentrated hydrofluoric acid on known titanium and titanium alloys, whereby
   - a yellow-green colored drop indicates that the unknown specimen is Ti-99,
   - a light brown-green colored drop indicates that the unknown specimen is Ti-8Al-1Mo-1V,
   - a dark brown-green colored drop indicates that the unknown specimen is Ti-6Al-2Sn-4Zr-2Mo.
   - a light green colored drop indicates that the unknown specimen is Ti-6Al-4V,
   - a dark green colored drop indicates that the unknown specimen is Ti-13V-11Cr-3Al,
   - a gray-green colored drop indicates that the unknown specimen is Ti-16V-2½Al, and
   - a black colored drop indicates that the unknown specimen is Ti-4Al-3Mo-1V.

10. A method for nondestructive spot testing a titanium alloy for the presence of aluminum, comprising:

(a) cleaning a test spot on the unknown titanium alloy specimen to remove any dirt, paint, grease, oxide or added metallic coating by use of a cleaning solvent, file, abrasive or the like,

(b) placing one drop of concentrated hydrofluoric acid on the clean metal surface of the specimen,

(c) after one minute adding three drops of concentrated nitric acid,

(d) after one minute, adding six drops of distilled water and transferring the resulting solution to a small test tube,

(e) adding 0.5 ml. of 6 M sodium hydroxide to the test tube and stirring with a glass rod until thoroughly mixed,

(f) adding an additional 0.5 ml. of 6 M sodium hydroxide and heating the test tube in a boiling water bath for three minutes,

(g) removing the tube from the bath and centrifuging at approximately 1725 r.p.m. for approximately five minutes,

(h) transferring three drops of the supernatant liquid to a spot plate depression and acidifying with six drops of 1 M acetic acid,

(i) adding two drops of an alizarin reagent, prepared by dissolving 0.06 gram of Alizarin Red S in 60 ml. of distilled water and observing the color of the solution in the spot plate depression after five-to-ten minutes,

whereby, a red-to-orange color indicates the presence of aluminum alloyed with the titanium while if no aluminum is present to react with the acidified solution of alizarin, the solution in the spot plate depression turns yellow.

11. The method of claim **10** and further including a semiquantitative test for aluminum wherein the intensity of the red-to-orange color is indicative of the aluminum present in the alloy and further comparing the color obtained in the spot plate depression with the color of known aluminum alloys in titanium as found on a standardized color chart.

12. A method of nondestructive spot testing a titanium alloy for the presence of molybdenum comprising:

(a) cleaning a test spot on the titanium alloy specimen by use of a cleaning solvent, file, abrasive or the like to remove any dirt, paint, grease, oxide or added metallic coating from the alloy,

(b) placing one drop of concentrated hydrofluoric acid on the cleaned spot and allowing it to react for one minute,

(c) adding one drop of concentrated nitric acid to the previous acid drop and after one minute,

(d) adding one drop of distilled water to the previous acid drops,

(e) transferring two drops of the combined diluted acid mixture to a spot plate depression,

(f) adding two drops of 10% potassium thiocyanate solution to the spot plate depression and stirring the various drops together,

whereby a brown color develops when the potassium thiocyanate is added and in the presence of molybdenum this brown color is transformed into a pink-to-violet color the intensity of which is proportional to the molybdenum content of the alloy.

13. A method of nondestructive spot testing a titanium alloy for the presence of molybdenum comprising:

(a) cleaning a test spot on the titanium alloy specimen by use of a cleaning solvent, file, abrasive or the like to remove any dirt, paint, grease, oxide or added metallic coating from the alloy,

(b) placing two–three drops of a test solution consisting of 25% concentrated nitric acid plus 5% concentrated hydrofluoric acid plus 70% distilled water, all by volume, on the cleaned metal surface and allowing it to react for two minutes,

(c) placing one drop of 6 M hydrochloric acid on a filter paper disc.

(d) transferring one drop of the test solution from the metal specimen to the center of the wetted fleck of the filter paper,

(e) adding one drop of 10% potassium thiocyanate solution and one drop of 25% sodium thiosulfate solution to the center of the wetted fleck,

whereby, a pink-to-violet color developing in the center of the wetted fleck confirms that molybdenum is present in the alloy and the intensity of the pink-to-violet color is proportional to the molybdenum content.

14. A method of nondestructive spot testing a titanium alloy for the presence of tin comprising:

(a) preparing a fluoroboric acid test solution by dissolving 28 grams of boric acid in 60 ml. of 48% hydrofluoric acid,

(b) cleaning a spot on the titanium alloy to remove any foreign material on the alloy,

(c) placing approximately ten drops of the fluoroboric acid test solution on the cleaned metal surface,

(d) after five minutes reaction time, gently flush the spot with distilled water to wash off the liquid acid, and

(e) drying the spot in a gentle stream of air to thereby leave a gray-to-black powdery precipitate on the metal surface if tin is present in the alloy.

15. The method of claim **14** wherein to confirm the presence of tin

(a) ten drops of concentrated hydrochloric acid are added to the gray-to-black powdery precipitate on the test spot and stirred with a glass rod,

(b) after five minutes of reaction time five drops of the acid solution are transferred to a test tube and diluted with five drops of distilled water,

(c) centrifuging the diluted solution at approximately 1725 r.p.m. for three to five minutes,

(d) pour off the clear liquid solution into a clean test tube,

(e) add three drops of a saturated mercuric chloride solution to the clear liquid,

whereby a white-to-gray precipitate formed at this point confirms the presence of tin with the amount of precipitate and the color intensity thereof being indicative of the amount of tin in the alloy.

16. A method of nondestructive spot testing a titanium alloy for the presence of vanadium comprising:

(a) cleaning a test spot on the titanium alloy to remove any foreign material on the alloy.

(b) adding one drop of concentrated hydrofluoric acid to the cleaned surface,

(c) after one minute, adding three drops of 1:1 nitric acid to the hydrofluoric acid and, after one minute, stirring to mix the acids,

(d) adding an additional two drops of 1:1 nitric acid, if needed, to remove any brown color and thereby change the color obtained to a blue-green when there is a presence of a high content of vanadium in the alloy.

17. A method of confirming vanadium in a titanium alloy as in claim **16** wherein:

(a) two drops of the blue-green acidic solution obtained in step (d) of claim **16** are transferred to a spot plate depression,

(b) adding one drop of a α,α' dipyridyl reagent to the spot plate and stirring, said α,α' dipyridyl reagent being prepared by dissolving 1.2 grams of α,α' dipyridyl in 50 ml. of 0.1 M HCl,

(c) adding and stirring in, respectively, four drops of concentrated ammonium hydroxide and two drops of concentrated phosphoric acid whereby,

a pink-to-red color confirms the presence of vanadium with the color intensity being proportional to the quantity of vanadium in the alloy, while a white color at this point indicates that the suspected alloy is commercially pure titanium.

18. A method of nondestructive spot testing a titanium alloy for the presence of zirconium, comprising:

(a) cleaning a test spot on the test specimen to remove any foreign material on the alloy,

(b) placing six-to-eight drops of concentrated hydrochloric acid to the cleaned surface and heating gently over an open flame for several minutes,

(c) placing two drops of 2 M sodium hydroxide on a sheet of Whatman #3 filter paper,

(d) transferring one drop of the warm acid solution from the test specimen to the wetted surface of the filter paper,

(e) adding one drop of 0.1% alizarin solution to the center of the wetted fleck,

whereby, a red precipitate that does not dissolve in 1 M HCl confirms the presence of zirconium and the intensity of the red color and the quantity of precipitate obtained is indicative of the amount of zirconium present in the alloy.

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