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Research Article

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Gravimetric Determination of Percentage Zirconium in Cement Based Ultra Temp-516 Using Mandelic Acid

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Abstract The gravimetric determination of percent zirconium in cement based, Aremco sample such as Ultra Temp-516 has been investigated for mandelic acid.The Ultra-Temp 516 has been fused with potassium hydrogen fluoride (KHF_2) to dissolve the Zirconium (Zr) and expel silisium dioxide (SiO_2). Sulfuric acid (H_2SO_4) was added and the mixture was heated to drive off hydrogen fluoride (HF) and convert the potassium fluoride(KF) to potassium hydrogen sulfate ($KHSO_4$).This mixture is ignited to convert the $KHSO_4$ to potassium sulfate (K_2SO_4), thereby eliminating the last traces of F^-. The precipitate was filtered, ignited and weighed as percent zirconium oxide (ZrO_2).

Keywords Zirconium, Ultra Temp 516-Cement, Gravimetric Method, Mandelic acid

1. Introduction

Ultra-Temp 516, a new temperature ceramic-based adhesive manufactured by Aremco products, Inc., is now used to bond zirconia ceramics used in applications to 4400 ^oF. Ultra-temp 516 is a new. Single part, zirconia-filled, inorganic adhesive system for bonding zirconia components used in high temperature sensors and instruments to 4400 ^oF. Ultra-temp 516 demonstrates exceptional electrical, mechanical and thermal properties. Its dielectric strength is 250 volt/mil; volume resistivity is 108 ohm-cm; and torque strength is 8.6 in-lbs. Due to its chemistry Ultra-temp 516 will not outgas under ultra-high vacuum (UHV), a common requirement for many industrial processes such as semiconductor manufacturing. Ultra-Temp 516 also provides excellent chemical, moisture and thermal shock resistance. Ultra-Temp 516 is applied easily using a syringe, brush or automatic dispenser. Because it is water-based, cleanup is accomplished quickly using warm water and soap. Full curing is achieved by heating up to 700 ^oF for 1-2 hours. Ultra-Temp 516 can be used to bond a wide range of additional materials including alumina, beryllium, silicon, graphite, Inconel, molybdenum, nickel, tantalum, titanium, and tungsten [1].

2. Materials and Experiments

Equipment/*Reagents:*

- Platinum crucible
- Bunsen Burner
- H_2SO_4 (1:1) Test Solution
- Ultra-Temp 516 is obtained from Aremco, New Jersey USA.
- 20% HCl Test Solution
- 15 % Mandelic acid Test Solution (Mandelic acid, 99.0% Sigma-Aldrich, lot# WXBB7099V
- Potassium hydrogen fluoride (KHF_2) Alfa Aesar, 98%, lot# X28A061

- Millipore filter (47 mm, 0.45µm)

2.1. Determination of Zirconium Oxide (ZrO_2)

Procedure:

A 0.500 g sample was grinded, was dried at 110 degrees C for 1 hour, and was mixed with 5 g of KHF_2 in a platinum crucible. This mixture was covered and heated very slowly over the Bunsen burner for a few minutes until the mixture was completely melted. The crucible heated the fusion until it was orange to convert KHF_2 to KF. The platinum crucible was removed from the flame and rotated so that the melted mixture was solidified on the sides. The crucible was allowed to cool. 10 mL 1:1 H_2SO_4 was added into the platinum crucible. The platinum crucible was placed on a hotplate where it got medium heat. The water in the mixture was evaporated off at a slow rate without boiling to ensure the mixture does not have form and to ensure that some of the sample is not lost by splattering. The evaporation took place four 4 hours, during which time it can be left unattended. When the mixture reached the point where it could be heated foaming, the crucible was picked up carefully with tongs and was cautiously heated over the Bunsen burner to evaporate the excess H_2SO_4. The mixture was set on a hotplate when it started to boil, and this was repeated for all of the crucibles. When the excess H_2SO_4 boiled off, the fusion was heated until it was orange to convert $KHSO_4$ to K_2SO_4.

The melt showed a tendency to crystallize when the conversion was completed. The cool crucible and cover were placed in a 400 mL beaker, and digested with 150 mL of 20% HCl solution. After removal and washing of crucible and cover, dissolved mixture was rinsed with water into the beaker. A 50 mL of 15% mandelic acid was added to the hot sample solution. Precipitation of zirconium started in a minute or two. The solution was allowed to stand overnight.

Note: *Owing to the low solubility of mandelic acid in 20% HCl some will crystallize out as the solution cools, however it will re-dissolve when the beaker is reheated. This same tendency prevents filtration through filter paper: Crystals form on the paper leaving is completely clogged. The following Procedure avoids this problem.*

The sample was reheated, then filtered the hot solution was filtered rapidly through a Millipore filter paper (47 mm, 0.45µm). This filtrate kept as much as of the precipitate in the beaker as possible. The precipitate and beaker washed three times with a wash solution prepared by dissolving 2g mandelic acid in 100 mL of 10% HCl [2]. The funnel was removed from the Millipore apparatus and rinsed any adhering precipitate back into the beaker. The precipitate was washed onto a 12.5 cm Whatman No. 40 paper. The filter paper was rinsed 10 times with the wash solution.

The filter paper was removed, folded loosely and set into a platinum crucible. The platinum crucible was placed on a wire gauze over a Bunsen burner with the flame turned down as low as possible. The heat was regulated so that the paper charred, but did not catch on fire. The wire gauze was replaced with a triangle and the crucible was ignited to burn off most of the carbons. The platinum crucible transferred to muffle furnace and heated at 900 ^oC for one hour, or until all the carbon was burned off. The crucible was cooled in a desiccator.

The ZrO_2 was brushed onto balance scoops and weighed (Table 3). The zirconium content was calculated from the original sample. The zirconium content of the original sample was calculated by the formula given below [2].

$$\%\ Zr = \frac{net\ \ ZrO2\ x\ 0.7403\ x \frac{Zr}{ZrO\ 2}\ x\ \%\ solids}{sample\ \ \ weig\ ht(g)}$$

3. Results and Conclusion

In this gravimetric test method, percent moisture was determined at the 70-75% (Table 1 and Table 2).The percent zirconium was determined and satisfactory results were obtained at the about 30% with mandelic acid level by reported procedure [2] (Table 3).

3.1. Determination of % Solid

Table 1: % Solid Determination

	Test 1	Test 2
Al dish (tare) (g)	1.2851	1.2843
Al dish + Wet sample(g)	8.1537	10.0540
Net wet sample (g)	6.8686	8.7697

After drying in oven at 105 °C for 1 hour

Table 2: % Solid Determination

	Test 1	Test 2
Al dish +dry sample(g)	6.3849	7.7616
Net dry sample(g)	5.0998	6.4773
% Solid	74.25%	73.86%
Average % Solids	73.925%	

% Solid=$\frac{\boldsymbol{net\ dry\ sample}}{\boldsymbol{net\ wet\ sample}}$x 100

3.2. Zirconium Oxide (% ZrO_2) and Zr determination

Table 3: Zirconium Oxide (% ZrO_2) and Zr determination

	Sample weight (g)	Crucible (tare) (g)	Crucible+ sample (g)	Crucible + ZrO_2 (g)	Net Zr %
Test 1	0.5110	4.8874	5.1708	0.2834	30.39
Test 2	0.5169	4.8874	5.1688	0.2802	30.00

$$\%\ Zr = \frac{net\ \ ZrO2\ x\ 0.7403\ x \frac{Zr}{ZrO\ 2}\ x\ \%\ solids}{sample\ \ \ weig\ ht}$$

Reference

[1]. Ultra-Temp 516 Zirconia Ceramics, Aremco. October 17, 2104. NJ.

[2]. John R. Sherman, International Testing Laboratories, Newark, NJ. May 30, 1997

[3]. R.S. Young "Chemical analysis in Extractive Metallurgy" Charles Griffin & Co. (London) pp384-385

[4]. Y.S. Su Anal-Chem, 37 (1965) pp 1067-1068.