NASA TECH BRIEF

Lewis Research Center

Rapid Method for Determining Nitrogen in Tantalum and Niobium Alloys

A commercially available instrument developed for the analysis of gases in steel has been adapted to rapidly determine the nitrogen content in tantalum and niobium alloys. The conventional method for determining nitrogen content, a modified Kjeldahl procedure, requires four to six hours; this new method requires less than four minutes.

Refractory alloys are promising candidates for skin materials for the Space Shuttle. Nitrogen and other interstitial elements are critical to the strength and corrosion resistance of refractory metals at high temperatures. Thus, it is important to determine nitrogen content, and to monitor changes in nitrogen content, in these alloys below the 100 parts-per-million level. The method most often used is a modified Kjeldahl procedure consisting of chemical dissolution, distillation and spectrophotometric measurement which takes four to six hours to complete. Investigation of an inert gas fusion procedure resulted in the adaptation of a commercial instrument, developed to measure nitrogen and oxygen in steel, which gave comparable results in less than four minutes.

In this inert gas fusion procedure, the sample is heated to approximately 2973 K (4892°F) in a helium atmosphere in a single-use graphite crucible. A platinum flux is used to facilitate melting of the sample. The released gases are separated chromatographically and measured in a thermal-conductivity cell.

A comparison of nitrogen values by the inert gas fusion method and a modified Kjeldahl procedure has been made for two tantalum-base alloys and six niobium-base alloys. The designation and nominal composition of the alloys tested are: T-111 (Ta-8W- 2Hf), T-222 (Ta-10W-2.5Hf), B-66 (Nb-5Mo-5V-1Zr), Cb-752 (Nb-9.8W-2.7Zr), Nb-1Zr, C-129Y (Nb-10W-10Hf-0.1Y), WC-3015 (Nb-29Hf-15W-1Zr-4Ta), and FS-85 (Nb-28Ta-10W-1Zr).

The inert gas fusion method results are comparable to the modified Kjeldahl results. For the tantalum and niobium alloys tested, the standard deviation for the inert gas fusion method was 5 to 7 percent.

Advantages of the inert gas fusion method are:

1. Both oxygen and nitrogen are determined on the same sample specimen;
2. The elimination of the time consuming Kjeldahl dissolution, distillation, and spectrophotometric measurement steps; and
3. Reduction of interference by nitrogen compounds normally present in the laboratory atmosphere.

Notes:

1. Further information is available in the following report:
   NASA TM-X-3067 (N74-26032), Comparison of Inert Gas Fusion and Modified Kjeldahl Techniques for the Determination of Nitrogen in Niobium Alloys
   Copies may be obtained at cost from:
   Aerospace Research Applications Center
   Indiana University
   400 East Seventh Street
   Bloomington, Indiana 47401
   Telephone: 812-337-7833
   Reference: B74-10085
2. Specific technical questions may be directed to:
   Technology Utilization Officer
   Lewis Research Center
   21000 Brookpark Road
   Cleveland, Ohio 44135
   Reference: B74-10085

Patent Status:

NASA has decided not to apply for a patent.

Source: E.J. Merkle, J.W. Graab, and W.F. Davis
Lewis Research Center
(LEW-12237)

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