QUANTITATIVE OXYGEN ANALYSES FOR V-4Cr-4Ti ALLOYS

Y. Yan, D.P. McGann, D.L. Smith and H. Tsai (Argonne National Laboratory)

OBJECTIVE

The objective of this task is to perform quantitative oxygen analyses on V-4Cr-4Ti alloys from ANL's 832665 heat, the General Atomics (GA)'s 832864 heat and Japan's NIFS-1 heat, and to evaluate the effects of oxygen content on the mechanical properties of V-4Cr-4Ti alloys.

SUMMARY

A quantitative oxygen analysis has been performed for V-4Cr-4Ti alloys. The oxygen concentration is about 210 wppm for Japan's NIFS-1 heat, 324 wppm for ANL's 832665 heat, and 385 wppm for the GA's 832864 heat. Our experiment indicates that, after cutting, the oxygen analysis specimen should be pickled to remove the surface oxide.

EXPERIMENTAL PROCEDURE

Three V-4Cr-4Ti alloys: the heat 832665, the heat 832864 and the NIFS-1 heat from ~4-mm plate were cut into small pieces (weight: ~0.3 g) using a wet diamond saw. Specimen temperature was kept low with cutting water to reduce the oxidation produced during the specimen cutting. Before the cutting, the V-4Cr-4Ti plates from the heat 832665 and GA heat were annealed in a high vacuum (better then 3×10^{-7} torr) at 1000°C for 1 hour. After cutting, the original oxide patina on the surface was removed using pickling solution (40 ml HF, 40 ml HNO_3, 120 ml lactic acid, 100 ml distilled water). The specimens were rinsed thoroughly with the pickling solution for ~2 minutes. An ultrasonic cleaner with Acetone and then Ethanol was used for ~5 minutes to clean possible dusts/liquids attached on the specimens that were induced during sample preparation.

Oxygen analyses were performed by using a LECO oxygen determinator at Argonne National laboratory. The determinator is located in a glove box, and can be used to both irradiated and unirradiated specimens. According to the operation procedure, the determinator had been calibrated for the oxygen by a standard sample before the analysis was performed.

RESULTS

Pre-peak from the Surface Oxide

To evaluate the effect from the surface oxide, the oxygen analysis was performed on specimens from the heat 832665 under different sample preparation conditions: 1) as-cut specimens (no pickling); 2) after cutting, clean the specimens with Acetone/Ethanol in an ultrasonic cleaner; 3) after cutting, perform pickling and then cleaning the specimens with Acetone/Ethanol in an ultrasonic cleaner.

It has been found that, for the as-cut specimens of the heat 832665, there is a pre-peak of the oxygen (see Fig. 1), and the oxygen concentration is also higher than that in the specimens that were cleaned by Acetone/Ethanol in an ultrasonic cleaner. This indicates that there was some contamination on the specimen surface before ultrasonic cleaning. Our tests also showed that, after pickling and Acetone/Ethanol cleaning, the oxygen concentration is lower than both the as-cut and the as-cleaned samples. No pre-peak of the oxygen was observed in the pickled and then Acetone/Ethanol cleaned sample (see Fig. 2). Detailed analyses indicated that the total oxygen contribution from the surface contamination on the specimens induced during sample preparation could be as high as 20-30% of the total, depending on the sample preparation. Thus it is necessary to perform pickling and Acetone/Ethanol cleaning before the oxygen test is performed.

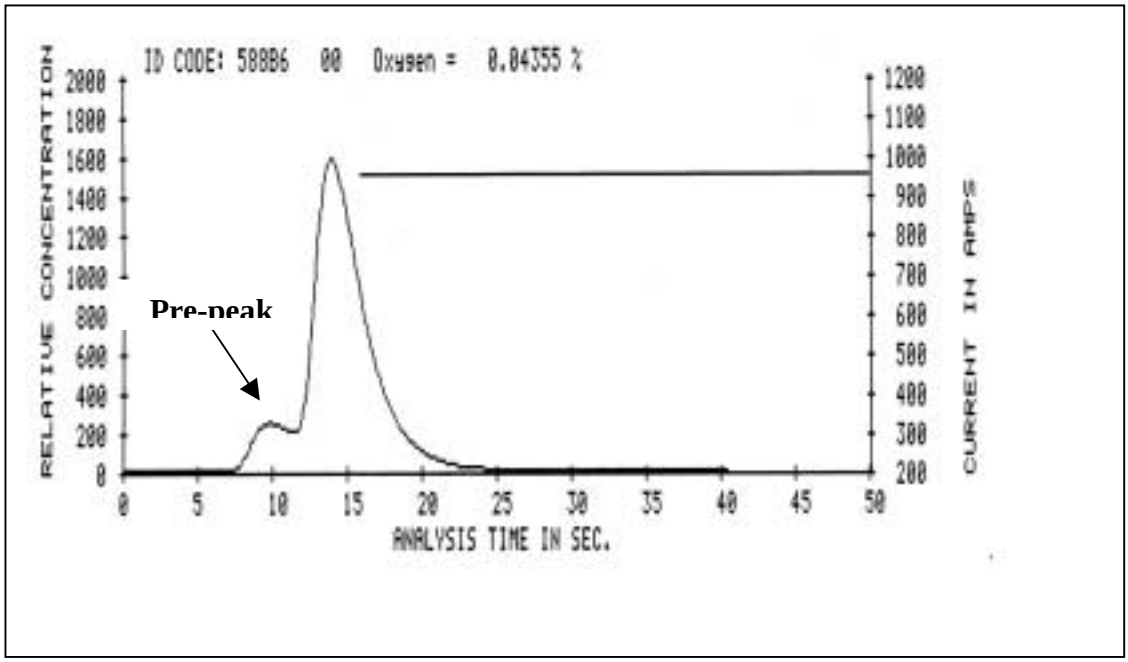

Fig. 1. Oxygen analysis for the as-cut specimen of the heat 832665.

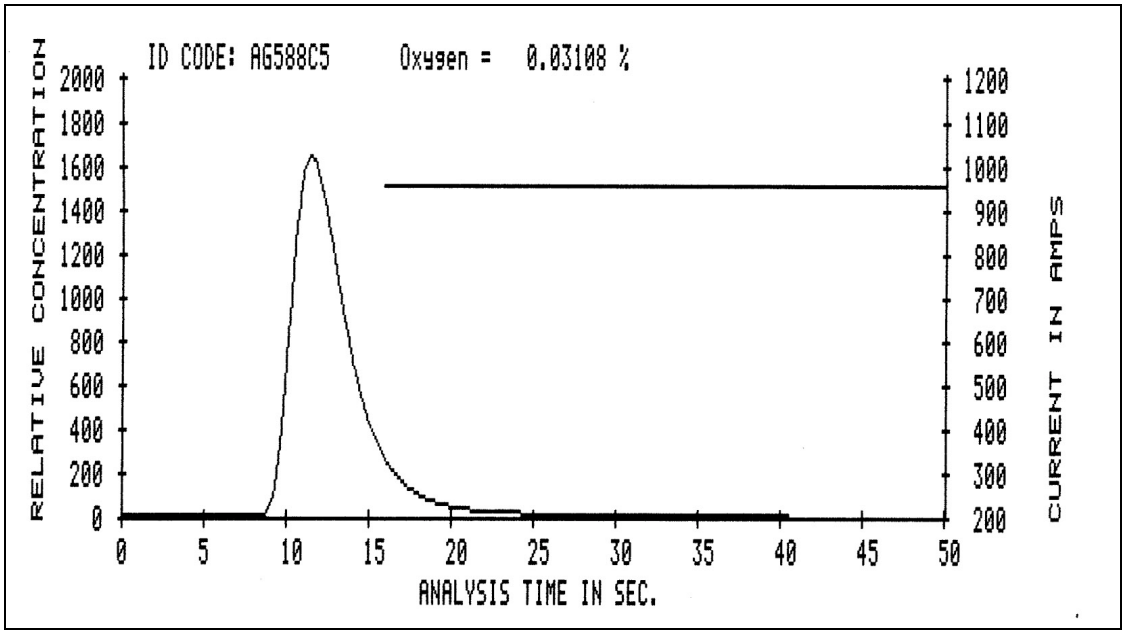

Fig. 2. Oxygen analysis for a specimen of the heat 832665. The specimen was pickled, and then cleaned by using Acetone/Ethanol.

ANL vs. Teledyne Results of Oxygen Analysis on V-4Cr-4Ti from the Heat 832665

To obtain a good statistical result, multi-tests were conducted for the V-4Cr-4Ti alloys from the different heats. The results of oxygen analysis on the 4-mm thick plate from heat 832665 are given in Table 1. The chemical analysis for oxygen determined by Teledyne Wah Chang on the ingot is included in the table for comparison. Our analyses indicated that the O content for the 832665 is about 324 ppm with a standard deviation ~3.4%, which is consistent with the Teledyne's result 310 ppm for the alloy ingot.

Table 1. Oxygen analysis on V-4Cr-4Ti from the heat 832665 (annealed plate)

ANL Data (4-mm plate)			TWC Data (ingot)		
Specimen ID	Weight (g)	Oxygen (%)	Specimen ID	Weight (g)	Oxygen (%)
588C5	0.3750	0.0311	1	N/A	0.0280
588C6	0.4232	0.0332	2	N/A	0.0360
588C7	0.2281	0.0315	3	N/A	0.0290
588C8	0.3303	0.0337			
Average		**0.0324**	**Average**		**0.0310**

ANL Results of Oxygen Analysis on V-4Cr-4Ti from the NIFS-1 heat and the GA heat 832864

Table 2 summarizes our results of oxygen analyses on V-4Cr-4Ti alloys from Japan's NIFS-1 heat, the GA's 832864 heat, and ANL's 832665 heat. It was found that the oxygen content of the Japanese heat NIFS-1 is lower than the heat 832665 and GA heat, and the GA heat has the highest oxygen content among these three heats in the ~4-mm plate. This consistent set of oxygen analyses can be used to evaluate the effects of oxygen on the differences in the Charpy impact and tensile properties of the V-4Cr-4Ti alloys. The ductile-to-brittle-transition temperature (DBTT) for all three heats of this alloy annealed at 1000^{o}C is below - 190^{o}C, indicating that the variations in the oxygen content at these levels has little effect on the impact properties [1, 2].

Table 2. ANL result of oxygen analysis on V-4Cr-4Ti alloy specimens

Heat	**Average Oxygen (%)**
NIFS-1	0.0210
GA	0.0385
832665	0.0324

FUTURE ACTIVITIES

The fracture surface of selected specimens after the impact test will be examined by scanning electron microscopy to delineate the fracture mode. Microstructure characterization on these three heats is underway and will be reported in the future.

REFERENCES

1. Y. Yan, H. Tsai, A. D. Storey, D.L. Smith, and Z. Xu, Microstructural characterization and impact properties of V-4Cr-4Ti Laser Weldments**,** Fusion Materials Semiannual Progress Report DOE/ER-0313/28, June 2000.
2. Y. Yan, H. Tsai, D.P. McGann, and D.L. Smith, Impact Properties of V-4Cr-4Ti alloy from the GA's 832864 heat, (this progress report).