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Development of titanium alloys for hydrogen storage

Abdul, Jimoh Mohammed

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ABSTRACT

The thesis investigated the effect of partial substitution of Cr or Ti with 2-6 at.% Fe, or 0.05-0.10 at.% Rh/Pd on the structure, hardness, corrosion behaviour and hydrogen storage characteristics of an arc-melted $Ti_{35}V_{40}Cr_{25}$ at.% alloy. The effects of an annealing and a quenching heat treatment on the properties were also investigated.

Melting of the eight alloys was done in a water-cooled, copper-hearth arc melting furnace under an argon atmosphere. Each of the eight ingots was cut into three: one as the as-cast sample and the other two separately quartz-sealed and loaded in two batches in a heat treatment oven and heated to 1000 °C for 1 hour. The first set of quartz tubes were immediately removed and broken in cold water to quench the alloy, hence locking the microstructure. The second batch was loaded into the furnace, heated to 1000 °C for 1 hour and then slowly furnace-cooled. The alloys (as-cast and heat treated) were characterised for phase identification using optical microscopy, X-ray diffraction (XRD), and scanning electron microscopy (SEM) with Electron Diffraction X-ray Spectroscopy (EDS) using an Oxford system. Thermo-Calc software was used to model the phases using the Solid Solution 4 and Titanium 3 Databases. The hardness values (under a 2 kg load) of all samples were recorded. Potentiodynamic corrosion tests were performed in 6M KOH at 25 °C, and Tafel curves were recorded from -1.4V to -0.2V with a scanning rate of 1mV/sec. A Sievert's apparatus was used for pressure composition temperature (PCT) measurements at 30, 60 and 90 °C.

All the alloys contained a primary bcc (V) phase. The secondary phases were a combination of αTi, $Ti(Cr,V)_2$ Laves phases (C14, C15 or C36) and a minor ωTi phase. The cell volume of the primary (V) phase decreased with addition of Fe and 0.05 Rh but increased with 0.1 Rh and Pd.

The hardness of the base alloy increased with additions of Fe and 0.10 at.% Pd, but decreased with additions of Rh and 0.05 at.% Pd. Additions of Rh, Pd and 2 at.% Fe decreased the corrosion rate, while additions of 5 and 6 at.% Fe increased the corrosion rate. The reversible hydrogen storage capacity (RHSC) of the base alloy, otherwise known as useful capacity, was enhanced with addition of Pd and Rh, but decreased with Fe addition.

Both annealing and quenching increased the hardness of the 0.05 at.% Rh and all the Fe containing alloys. Heat treatment decreased the hardness of the base alloy, both Pd alloys and the 0.10 at.% Rh samples. Quenching decreased the hardness of the 0.10 at.% Rh and both Pd-containing alloys.

The corrosion rate of the 0, 5 and 6 at.% Fe, 0.05 at.% Rh and the Pd-containing alloys decreased after annealing.at.% FeThe rate increased after annealing the 2 at.% Fe and 0.10 at,% Rh samples. The as-cast sample containing 2 at.% Fe had the lowest corrosion rate (0.0004 mm/y) and the quenched 6 at.% Fe was the least corrosion resistant sample with a corrosion rate of 0.037 mm/y.

The quenched 5% Fe alloy had the highest hardness (460 MPa), while the annealed 0.10 at.% Rh sample had the lowest (388 MPa).

The quenched 0.05 at.% Pd sample had the highest RHSC (2.28 wt%) while the lowest RHSC of 0.44 wt% was observed in the as-cast 2 at.% Fe sample.

Annealing improved the RHSC of all samples except the base $Ti_{35}V_{40}Cr_{25}$ and 6 at.% Fe alloys, while quenching was detrimental to RHSC of all the samples but the 2 at.% Fe, 0.05 at.% Pd and 0.10 at.% Rh alloys.

Increasing the addition of palladium from 0.05 to 0.10 at.% Pd showed no significant improvement on RHSC of the base alloy, thus addition of 0.05 at.% Pd would be sufficient. The RHSC of the annealed 0.05 Rh alloy (2.25 wt% H) was close to the value of the 0.10 at.% Pd, so rhodium could be considered as an alternative to the quenched 0.05 at.% Pd. The RHSC was 1.56, 0.44, 0.75 and 0.68 wt% for 0, 2, 5 and 6 at.% Fe as-cast alloys respectively. Although the 2 at.% Fe alloy had the lowest RHSC, it could find its application as electrode in 6M KOH solution electrolyte because of its low corrosion rate.