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Study of Pyrotechnic Disc Materials in Thermal Batteries by X-Ray Technique

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In this investigation, a comparative X-ray study for the pyrotechnic disc of the assembled cell has been carried out. The results were compared with those issued by the American Society for Testing Materials (ASTM). For this, the X-ray diffraction and the linear spectrum for this diffraction, belonging to the mixture used in industrialization of pyrotechnic disc of the thermal cell before ignition, has been accomplished. Peaks belonging to the fuel, Fe and the oxidizer, $KClO_4$, components were developed, indicating that the two components were incorporating in the composition of the pyrotechnic disc. Furthermore, another comparison for the X-ray diffraction values and the linear spectrum of this diffraction, for the thermal mixture after ignition, was brought about. Peaks pertaining to Fe_2O_3, KCl and ZrO have emerged, demonstrating that oxidation and reduction processes have taken place, where the mentioned products were formed by the reaction among Fe, Zr and $KClO_4$. The formation of ZrO revealed that Zr included in the composition of the active disc could be detected before ignition due to its low concentration.

Key Words: Thermal batteries, Thermal cells, X-ray, Pyrotechnic disc.

INTRODUCTION

Thermal batteries are primary reserve batteries utilizing an electrolyte which is a non-conductive solid at ambient temperatures. A thermal battery is characterized by having a very long shelf life which exceeds ten years without any imperfection in performance, requiring no maintenance during storage prior to use and thus allowing it to be permanently installed in an equipment[1]. Furthermore, it can be reliably operated after storage at temperatures of the range –54°C to 71°C. Additionally, it can be activated very rapidly. Once activated, the thermal battery provides electric power extending from a few seconds up to more or less 1 h. These characteristics nominate the thermal battery to be utilized worldwide in a number of military applications over wide ambient temperature ranges and

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severe dynamic environments. A thermal battery is activated by first activating an ignitable pyrotechnic heat source, which gives rise to the melting of the electrolyte thereby activating the battery cells.

Because the thermal battery may be subjected to a wide temperature range during storage, it is difficult to predict the ambient temperature at the time of activation and therefore the quantity of heat which will be required to melt the electrolyte and activate the cells in the battery. Due to the serious nature of the appliances which utilize the thermal batteries, namely the military equipments such as decoys, bombs, rockets, missiles, torpedoes and the emergency power systems like those existing in submarines and aircrafts[2], multiple heat sources are employed for a thermal battery[2].

In one configuration, the cell stack contains the primary heat source and a secondary heat source is wrapped around the thermal insulation surrounding the cell stack. The heat generated from the secondary source lessens the temperature gradient through the thermal insulation from the interior of the cell stack to the exterior environment. So the cell stack cooling rate is decreased leading to lengthening the active life[2]. The secondary heat source is generally a zirconium/barium chromate ceramic fibre heat paper[2–6].

In the second configuration, the thermal insulation surrounding the stack is rounded by a wrapped resistance heating element or wire. Power to this heating element may be supplied to warm a cold-stored battery before use. Otherwise, the thermal battery may be configured to provide the heating element with energy pulse during discharge of the thermal battery, hence diverting a fraction of the thermal battery output for heating purposes. As mentioned before, heat from the secondary source reduces the temperature gradient, thereby reducing the cell stack cooling rate and extending the battery's active life[2].

Previously[3], the effect of the composition of the pyrotechnic disc upon the efficiency of the thermal cell has been considered. It showed that, in the absence of the activating fuel, the mixture composed of 88% Fe and 12% $KClO_4$ was the most active, where it gave a voltage[5] of 1.82 V at 58 s. This result was referred to the increment in the thermal disc glow which offered the least heat content leading to the high voltage, 1.82 V. The results also demonstrated that using activating fuels such as Mg, Al or Zr led to some slight explosions and detonations, which gave rise to irregularity of the disc ignition. Among these, Zr was found to be the best.

The present project aimed to accomplish a comparative X-ray investigation for the components of the pyrotechnic disc of the assembled cell, before and after running the thermal cell, by measuring the diffraction values and plotting the linear spectra for the components and comparing the results with that issued by the American Society for Testing Materials (ASTM).

EXPERIMENTAL

All the used chemicals were purchased from BDH, E. Merck or Fluka, with purities > 95%. Preparation of the molten salts as well as storing the components of the thermal cell were conducted inside a dry box flushed with argon.

The chemical compounds resulting from electrochemical reaction of the pyrotechnic disc in the thermal cell have been identified *via* the use of X-ray diffraction instrument. The procedure was described before[7].

Measurements were performed by using X-ray diffractometer (PW 1840, Philips). From the X-ray spectrum scheme, the amplitude-d values for the pyrotechnic disc components were calculated by using Bragg's equation. As no crystalline forms with identical amplitude d-values can exist, distinction between crystalline materials in a sample is possible by calculating the amplitude d-values for them, without the necessity to separate these substances from each other. The amplitude d-value was compared with that reported by ASTM data[8].

RESULTS AND DISCUSSION

Table-1 and Fig. 1 demonstrate the X-ray diffraction and the linear spectrum for this diffraction, belonging to the mixture used in industrialization of the activating disc of the thermal cell before ignition. Peaks belonging to Fe and $KClO_4$ have arisen. This spectrum was obtained to recognize the change in the mixture, before ignition.

TABLE-1
A COMPARISON OF THE X-RAY DIFFRACTION FOR THE MIXTURE USED IN INDUSTRIALIZATION OF THE ACTIVATING THERMAL DISC BEFORE IGNITION

d-Value for the diffraction of the component (Å)	The products	d-Value for the diffraction of the standard compound (Å)
3.500	$KClO_4$	3.490
2.062		2.063
1.871		1.870
2.120		2.118
2.025	Fe	2.026

Table-2 and Fig. 2 reveal a comparison for the X-ray diffraction values and the linear spectrum of this diffraction, for the thermal mixture after ignition. New peaks have emerged, pertaining to Fe_2O_3, KCl and ZrO, indicating that oxidation and reduction processes have taken place, which gave rise to the quick blaze of the mixture. The proposed reaction is:

$$2Fe + Zr + KClO_4 \rightarrow Fe_2O_3 + ZrO + KCl$$

From Tables 1 and 2 the close similarity of the d-values for the diffraction of the products obtained from the thermochemical reactions at the pyrotechnic disc with that of the standard analogue reported by ASTM is clearly noticed[8].

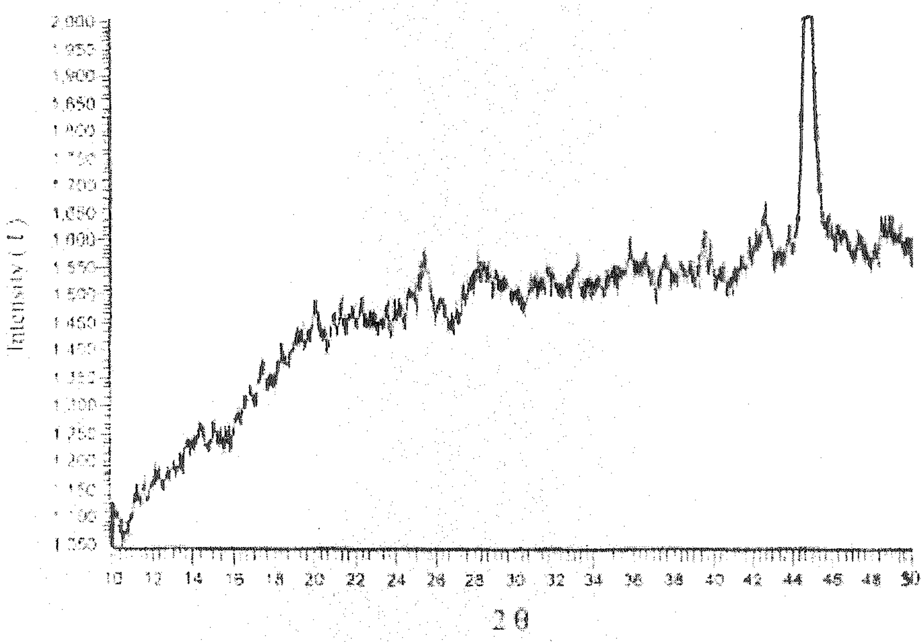

Fig. 1. Linear spectrum for the X-ray diffraction for the mixture used in industrialization of the activating thermal disc before ignition

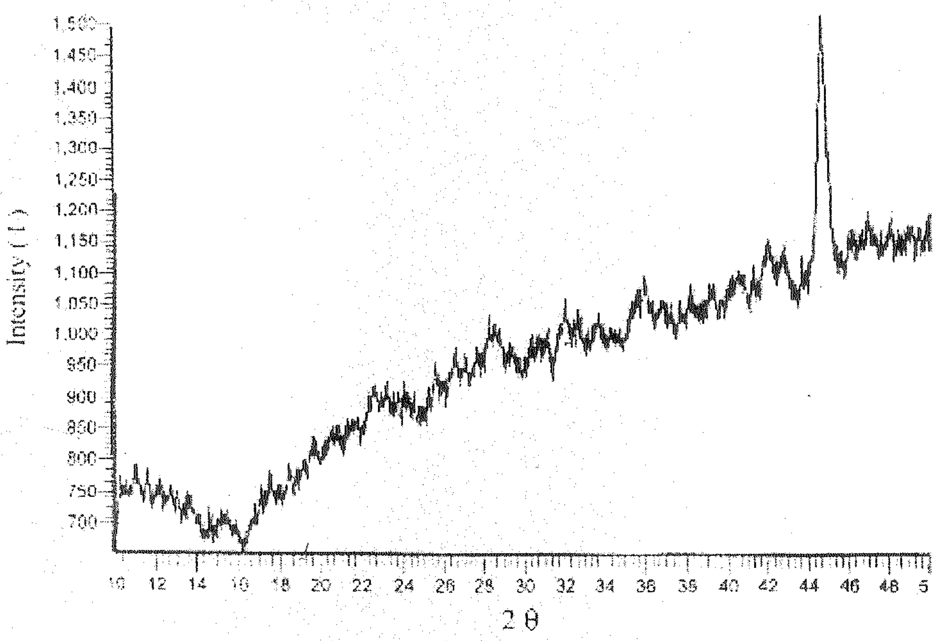

Fig. 2. Linear spectrum for the X-ray diffraction for the mixture used in industrialization of the assembled thermal disc after ignition.

TABLE-2
A COMPARISON OF THE X-RAY DIFFRACTION FOR THE MIXTURE USED IN INDUSTRIALIZATION OF THE ASSEMBLED THERMAL DISC AFTER IGNITION

d- Value for the diffraction of the component (Å)	The products	d- Value for the diffraction of the standard compound (Å)
2.080	Fe_2O_3	2.089
2.665	ZrO	2.670
2.225	KCl	2.224
3.154		3.150

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