THE MEASUREMENT OF SLIGHT WEAR OF ENGINEERING PLASTIC COMPOSITES USING NEUTRON ACTIVATION ANALYSIS

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ABSTRACT

In this paper, an experimental method was described for measuring very slight wear of polymer by using neutron activation analysis (NAA). By means of adopting appropriate neutron flux suitable experimental parameters and measuring technique, the method was performed under the condition without any effect upon the mechanical properties of wearing. The sensitivity of the wear measurements reached the order of magnitude of 10^{-7}g for engineering plastics containing about 1 wt% of zinc. The comparison could be made rapidly between different materials with different resistance to wear. The results were free from the influence of vibration of test systems, elastic deformation of materials, media and moisture absorption, and etc. Combined with autoradiograph, the present technique can be used to investigate the backtransfer of ground debris of metallic fillers from the counter part to the specimen. A multifunctional plane wear testing machine was set up for obtaining wear data and friction coefficient. The present work provides a new, rapid and sensitive measuring technique and also a screening method for the wear of polymers.

Keywords: Neutron activation analysis Plastic composites Wear

I. INTRODUCTION

Currently, conventional methods available for wear measurements are gravimetric and volumetric methods. It is always the case that because of the interferences of moisture absorption, thermo- deformation and the roughness of a specimen surface, especially polymers the measurements failed even if a sensitive electric induction apparatus is used. A specimen could only be tested after a wear distance of a number of kilometers. Electric measurements, such as electric capacity or induction are affected by media, deformation, vibration and friction heat in testing systems. All these factors cause difficulties for dynamical and accurate measurement of the wear of polymers.

Neutron activation analysis (NAA) is advantageous over conventional techniques to measure the wear of polymeric materials. 1) The results are free from the influences of vibration, working medium, deformation, or high temperature caused by the friction. They are also independent of the moisture absorption, elastic deformation and thermo- deformation of the specimen itself. 2) The measurements can be continuously monitored in situ without taking off the specimen from its operation. 3) By using autoradiograph, it is possible to study the metallic fillers that transfer back to the

polymer composite from the wearing counterface. This is uncapable with other methods. 4) The measuring technique is rapid and sensitive. The sensitivity depends upon the composition of metallic elements in the polymer and the neutron flux it bears. a detectable quantity of wear amount is 10^{-6} g in general. It takes only a few days to complete an NAA wear test instead of several weeks or even longer with the other methods.

The present work aimed at the application of NAA to measure very slight wear of PTFE- based polymer composites.

II. EXPERIMENTAL

1. Specimen

1) Specimen preparation The wear- resistant PTFE band was stuck on a glass fibre base plate and ground on a grinding machine to get a smooth surface, so as to minimize the effect of roughness on test results.

2) Calibration samples High purity zinc powder (99.999%) was used as calibration samples. 0.1 g zinc powder was weighed with a balance of 10^{-4} g sensitivity and sealed in a quartz tube. The calibration samples and specimens were irradiated at the same time in a reactor. Conventional chemical treatment was used to obtain standard calibration sources for different measuring sensitivities (10^{-5}, 10^{-6} and 10^{-7} g.).

2. Neutron irradiation

Tests showed that PTFE was readily broken into pieces when a total neutron flux N was greater than $2.1\times10^{17}/cm^2$. Therefore, a total neutron flux of $N=3.6\times10^{16}/cm^2$ was finally selected for irradiation. Table 1 lists the neutron activation parameters.

Table 1

Neutron activation parameters

Materials	PTFE and its composite
Total activation time	8h
Thermal- power	68000 kW
Total thermal- neutron flux	$3.6\times10^{16}/cm^2$
γ flux	2.46×10^4C/kg
Specimen chamber	11DS
Activation temperature	<100°C

3. Wear tests

The dimension of the specimens used was 3.5×7 cm^2. The specimens were made of

Table 2

Specific weights and zinc contents (wt%) of the specimens

Specimens	TF	SF	FT	TSF (1)	TSF (2)	JC- 20
d (g/cm^3)	2.64	2.45	1.8	2.76	2.93	2.8
Zn (wt%)	1.43	1.2	1.72	1.94	1.88	1.97

polymer based materials with various wear- resistances. For each material three specimens were tested. The specific weights and zinc contents (wt%) of the specimens relevant to the wear tests are listed in Table 2.

Testing condition The specimen was wear- tested under a normal stress of 54.88 $\times 10^4$ Pa. The counterpart was made of cast iron. Its surface roughness was estimated at 1.6- 0.8μ m, No. 20 machine lubricant oil was used for lubrication. To get homogeneous wear, the working surface was assured to keep flat in each test. The tests were performed on a plane wear testing machine. The speed of the working stage was set at $v = 6$m/min for this study. After a wearing distance of 1 km, the mixture of oil and ground debris was filtered and dried for counting measurement. The grain diameter of the filler was greater than 0.075 mm.

a)Reduction of thickness (R_t) $R_t = (t_b - t_a)/t_b \times 100\%$, where t_b and t_a are the thicknesses of the specimens before and after compression, respectively. R_t reflects the load capacity of the materials. The results of load capacities obtained on a compression testing machine are shown in Table 3. Different loads were applied to the specimens with a surface area of 1 cm^2. The load was kept for 5 min for each test, followed by measuring the compressed deformation of the specimen. The tests were conducted on the working stage of 9.8×10^6Pa.

Table 3

Load capacity

Force (N)	Thickness of PTFE before activation (mm)	Thickness of PTFE after activation (mm)
50	2.772	2.672
100	2.770	2.672
150	2.765	2.672
200	2.750	2.672
250	2.740	2.672
300	2.720	2.672
400	2.700	2.672
500	2.675	2.672
600	2.642	2.672
700	2.620	2.662
800	2.595	2.662
900	2.565	2.658
1000	2.530	2.656
Total compression	0.244	0.016
R_t under the force of 1000N	8.8%	0.6%

b)Friction coefficient The friction coefficient f for the plane wear testing machine was worked out. The results are given in Table 4.

c) Wear resistance Testing conditions: The tests for wear resistance were carried out on an Amsler testing machine. The counterpart was a Cr plated disc with the

Table 4

The data of friction coefficient *f* obtained from the plane wear testing machine

Speed (mm/min)	PTFE before activation	PTFE after activation
50	0.06077– 0.068	0.069
100	0.0518– 0.068	0.069
400	0.0507– 0.072	0.0582
1000	0.0536– 0.079	0.0636

surface roughness of 0.8μ m; $M=0.98$ Nm; $P=245$ N; rotation speed = 200r/min. The testing time was 10 min for each test. Table 5 shows the results of wear resistance.

Table 5

Wear resistance

Wear (mm)	PTFE before activation			PTFE after activation		
Width of the wear mark	10	10	10	7	5	6
Depth in the middle of the wear mark	0.49	0.69	0.70	0.28	0.19	0.21

d) Compressive strength and compressive modulus The experiments for testing compressive strength and compressive modulus were performed on INSTRON 1195 electronic testing machine. The size of specimen used was 10× 10× 20 mm, and the cross head speed for applying compressive force was 5 mm/min. The results are shown in Table 6.

Table 6

Compressive strength and compressive modulus

Property	PTFE before activation		PTFE after activation	
Compressive strength	2.695×10^7	2.656×10^7	2.264×10^7	2.234×10^7
Compressive modulus	11×10^8		11.8×10^8	

4. Activation measurement

β – rays from the irradiated samples were counted by a FJ– 2603 low– level α , β counting system. The background of β – rays was lower than 0.2cpm. By means of autoradiograph,the back– transfer of metallic filler from the counterface to the polymers was observed. The observation was made in following procedure. After the wear test of neutron activated specimen, in which some of the radioactive ground debris were left on the work stage, an unactivated specimen was tested on the same stage for the same duration. Then, the unactivated specimen was gently cleaned and dried. It was tightly pressed against an X– ray film to be exposed. Fig.1 shows a back– transfer autoradiograph in which the white spots indicats the

Fig.1 A typical view of autoradiograph showing back–transfered metallic filler in ground debris

metallic filler in the ground debris back- transfered from the counterface.

Ⅲ. RESULTS AND DISCUSSION

1. The results of wear tests

Tests on the standard calibration sample showed that we were able to achieve a sensitivity of 1.032×10^{-7}g/count. Assuming N as the counts of ground debris, the amount of wear $W=bN$ and the wear thickness $h=w/sdp$, where s is the wear area of the specimen in cm^2; d is the specific weight (g/cm^3) and p is the zinc content in wt%. In Table 7 are the results derived from TF samples. N100s indicates the counts of ground debris in 100s; N is an average value of the counts: k is the number of repeated tests (3 times); σ_N is the standard error of average counts; $N\neq\sigma_N$ is the wear result (the confidence is 68.3%); v is the relative error and h is the wear per kilometer. Table 8 shows the results obtained from the specimens made of other materials.

Table 7

Measured data and calculated wear from wear tests for TF material

Material	N 100 s	N	$\sigma_N=N/K$	$N\pm\sigma_N$	$v=\sigma_N/N$	h (μ m)
TF1	105,105,100	103	5.8	103±5.8	5.6%	0.12±0.01
TF2	99,100,99	99	5.7	99±5.7	5.7%	0.11±0.01
TF3	101,99,99	100	5.8	100±5.8	5.8%	0.12±0.01

Table 8

Measured data and calculated wear from the materials other than TF

Materials	FT_1	FT_2	FT_3	TSF (2*)$_1$	TSF (2*)$_2$	TSF(2*)$_3$	SF1	SF2	SF3
Wear rate	0.1±	0.09±	0.1±	0.23±	0.24±	0.23±	0.37±	0.36±	0.36±
(μ m/km)	0.01	0.01	0.01	0.02	0.02	0.02	0.01	0.01	0.01
Materials	JC- 20_1	JC- 20_2	JC- 20_3	TSF (1#)$_1$	TSF(1#)$_2$	TSF(1#)$_3$			
Wear rate	0.23±	0.25±	0.23±	0.6±	0.7±	0.6±			
(μ m/km)	0.02	0.02	0.02	0.05	0.05	0.05			

2. Discussion

a) As the method is based on activating metallic elements in polymers and counting the disintegrations of wear debris, the activity of a very small quantity can be detected with an appropreciate neutron irradiation. But, if the neutron flux is too high the polymer will become cross- linked or degraded. For this reason, an important procedure in NAA wear tests of polymers is to select the neutron flux. The total neutron flux used in the present work made it possible to reach the sensitivity of 10^{-7} g. The irradiation changed little of the load capacity, friction coefficient and compressive strength of the PTFE but increased the wear- resistance slightly. Although it was noted that the tensile strengths of the materials were decreased to some extent by the activation, it had little effect upon the results of wear tests.

b) The PTFE band of the polymer used for wear- resistant sliding guide was

composed of C, Cu, F, Cr, Sn and Zn. And the wear- resistant coating consisted of C, H, O, F, N and Si. Of all these elements, F, N and O become ^{20}F, ^{16}N and ^{19}O respectively after neutron activation and would be cooled out easily because the corresponding half lives of those radioactive elements are only 11, 7.4 and 29 s, respectively. The neutron capture cross section of C and H was so small that the radioactivity of ^{3}H and ^{14}C produced by $3.6\times 10^{16}/cm^2$ neutron flux was almost zero. Under neutron activation, Cr and Sn become ^{51}Cr and ^{113}Sn which do not emit β - rays. Thus Cr and Sn was precluded from interference when only the β - rays of the ground debris were detected far as Si and Cu are concerned, the half lives of ^{31}Si and ^{64}Cu are 2.62 h and 12.9h respectively. Just one week of cooling time would reduce the radioactivities to respectively 10^{-20} and 10^{-5} times lower. It was proper to choose zinc as a calibration element because the half life of neutron activated ^{65}Zn is 245 days, and the energy of its β - ray is 0.33 MeV.

c) In order to reduce the error of wear measurements, a low level system ($B_\beta \leqslant 0.2$ cpm) and multistandard sources were used. Calibrations were done with the standards and a curve was obtained by means of the least square method. The relative errors of the measurements were reduced by selecting appropriate length of measuring time and the number of measurements. The relative error in this work was between 3% and 5.8%, whereas it was about 10% with conventional methods to measure polymer wear.

d) NAA enables one to observe the back- transfer of ground debris by means of autoradiograph. A typical result is shown in Fig.1. There was only a few spots of metallic debris back- transfered from the counterface. This can be explained by poor adhesion of PTFE.

e) The technique is applicable to many other polymeric materials, such as polyethylene, polystyrene and which are more endurable than PTFE to high neutron dosages.

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