THE EFFECT OF COATING PARAMETERS AND ANNEALING ON THE CRUSHING STRENGTH OF TRISO COATED PARTICLES

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The crushing strength (load at fracture) of coated particles was measured by compression between soft metal anvils. The method requires no sample preparation and can be used as a quality control method as well as a valuable tool for comparing different coating conditions during the manufacture of TRISO particles. Batches of coated particles manufactured with different coating parameters were prepared and tested. Batches prepared under different conditions exhibited significant differences in crushing strength. Higher argon concentrations in the coater gas mixture resulted in higher crushing strength. Anomalies in the crushing strength of particles are related to defects and possibly residual stresses produced during coating. The influence of annealing at 1950°C on crushing strength was also investigated. The average crushing strength of batches of particles decreased with annealing. Different preparation methods showed a marked difference in the level of deterioration of the particles with annealing. Batches produced with 80% argon gas mixture at 1300°C showed the greatest reduction in crushing strength. An inverse correlation was found between the crushing strength and the uranium that is leached from batches. The particles with the lowest crushing strength would also be more susceptible to mechanical damage during handling and consequently also to leaching of the uranium during leach tests.

I. INTRODUCTION

High-temperature reactors are probably the most likely type of reactors to be constructed in the future.[1] Although there are differences in the designs of these reactors, the basic form of the fuel is always in the form of a coated particle. Because of the high temperatures of the fuel, the properties of the layers of the coated particle are fundamental to the safety of these reactors. In the case of TRISO particles, the surrounding layers act as a containment vessel for the fission products.

The TRISO fuel particle consists of a 500-μm UO_2 fuel kernel, which is covered with three types of isotropic layers. The layers consist of a porous carbon layer (buffer layer), an inner high-density carbon layer (IPyC), a SiC layer, and an outer high-density carbon layer (OPyC).

Because of the presence of three isotropic layers, the particles are known as TRISO particles. A number of these coated particles together with graphite and a resin are pressed and sintered into fuel elements for use in the reactor.

The function of the layers of a TRISO particle is to contain the fission isotopes under normal operating conditions and also when the spent fuel is stored. High integrity, assessed on the physical strength of these particles, is of vital importance. Over the last few decades the measurement of the strength has been a contentious issue.[2–5] Methods such as crushing between hard anvils, the brittle ring test, and others were mostly considered unsatisfactory and not representative of the stress to which the particles will be subjected to in practice. Most of the tests that were previously proposed also require substantial effort in sample preparation, which limits its use as a routine quality control test.
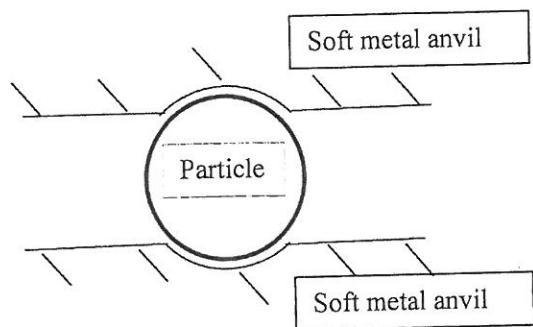
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Fig. 1. Diagrammatic presentation of the crushing test.

A recent publication[6] describes a test method that requires no sample preparation and also simulates the stress to which the particles are subjected in use. This method determines the crushing strength of a particle between soft metal anvils rather than testing between hard anvils that do not deform plastically during testing. Figure 1 shows a diagrammatic presentation of such a test at the stage at which a particle fails by fracturing.

There is a soft layer between the UO_2 kernel and the coated layers. Therefore, the coated layers can be approximated to a spherical shell. If the particle (shell) is strong enough and the anvils are soft enough, the particle does not fail initially when contact is made with the anvils. Instead, as the load increases, an impression is produced in the soft anvils. The spherical shell will deform elastically so that the diameter around the equator will increase. This is indicative of a circumferential tensile stress in the shell. The magnitude of the tensile stress can be calculated by means of finite stress analysis.[6] By using soft anvils, a large volume of the shell is subjected to tensile stress that simulates the condition when a particle is subjected to internal pressure. Failure of the particle then originates as a result of crack initiation and crack propagation at right angles to the applied stress. Experimental tests confirmed that fracturing does not initiate at the contact but rather in the layer of material between the anvils.[6] Anvils can also be too soft, in which case the particle will not fail but submerge completely in the anvils. Selection of anvils with suitable hardness is therefore important. Different materials have been investigated, and anvils with a Vickers hardness of 38 were selected for testing the particles.

In the case of nondeforming hard anvils, the maximum stress during testing develops at the point of contact. The stress in the shell at the point of contact is then of a bending nature in which only a very small volume of shell is subjected to tensile stress that can produce cracking and failure.

In this study, the testing method is used to determine the average crushing strength of batches that were prepared with different coating parameters. A variety of coating parameters have been employed in the Research Coater Facility (RCF). The crushing strength of these batches of particles was also compared with the crushing strength of TRISO particles produced using standard conditions.

The influence of annealing on the crushing strength of batches was determined[7] using the crushing test as described previously.[6] These results[7] were obtained from batches of zirconia-coated kernels that were prepared during the early commissioning of the coater facilities. These batches generally showed an increase in the crushing strength with annealing. Contrary to this,[2] it was found that there is a decrease in the crushing strength of batches of TRISO particles with annealing. It is extremely important for fuel particle modeling to quantify the effect of annealing on standard quality fuel particles. Some of the annealing tests were repeated using standard quality TRISO particles with the method described.[6]

Historically, it was found that there is always a small fraction of isotopes, formed by fission, that escape from the coated particles under operating conditions. During fuel fabrication, no quality control method can simulate the reactor conditions. Fuel elements could be in the reactor for a period of $\sim 2\frac{1}{2}$ yr at high temperatures. In contrast to this, quality control tests are done for a period of only 16 h. It is found that the results vary from the control tests on batches of coated particles, even for particles prepared under standard conditions. These batches of coated particles, which appear to be of the right quality, leak uranium when subjected to certain quality control tests.[8] Leach tests are the only tests that give an indication of the integrity of the layers. High leach results were attributed to damage due to mechanical handling in the past.[9–11] Weaker particles could be more vulnerable to damage during the fuel fabrication process. Therefore, it is important to investigate the link between particle strength and leach results.

II. EXPERIMENTAL FACILITIES

The Fuel Development Laboratories consist of four laboratories: a kernel production laboratory, a coater laboratory, a fuel sphere laboratory, and a quality control laboratory. There are two coaters in the coater laboratory.

The coaters used the same chemical vapor deposition process to coat particles. The coater consists of a vertical graphite reaction tube. The tube is heated by means of a graphite element. The temperature is controlled by pyrometer measurements on the outer surface of the reaction tube. A mixture of fluidization and reaction gases is injected into the bottom of the coater. The fluidization pattern of the particles can be described as a spouted bed.

II.A. Research Coater Facility

The RCF has been in operation since 2001. Initially, the purpose was to establish the technology to produce

TRISO coated particles. When the construction of a commercial coater facility started (~2006), the RCF became a research facility.

The facility was licensed to coat depleted or uranium of natural composition. Up to 1 kg of UO_2 kernels can be coated at a time. Normally, however, batches of 0.75 kg of UO_2 kernels were coated. The advantage of such a large RCF is that the results would be applicable to a commercial fuel coating facility.

In the facility, approximately 240 batches have been coated using a wide variety of coating parameters. After the initial development phase, variations in the standard coating procedure were introduced in order to establish trends.

II.B. Advance Coater Facility

The commercial size coater or the Advance Coater Facility (ACF) can coat batches up to 5 kg of UO_2 kernels. It was initially commissioned with zirconia and later with depleted UO_2 kernels. It was then licensed to coat a campaign of two batches of 9.6% enriched UO_2 kernels. Standard quality coated particles refer to batches of coated particles that are of the same quality as these two batches.

III. SAMPLING AND TEST METHODS

III.A. Sampling and Definitions of Sample Types

Samples were taken by decreasing the flow of gas through the coater after layers had been deposited, thus allowing a sample (between 5 and 20 g) to drop into a sample container at the bottom of the coater. The sample container is lined with a carbon foil to reduce the impact of the particles with the bottom of the container.

Three types of samples were taken. An IPyC sample consisting of kernels that were coated with a buffer and an IPyC layer (this is equivalent to a BISO particle, except that the IPyC layer is thinner than in a BISO particle). A SiC sample consisted of particles that had a buffer, an IPyC, and a SiC layer. The TRISO sample consisted of particles with an additional OPyC layer.

III.B. Crushing Strength Test

Particles were randomly selected from a sample and subjected to crushing between soft metal anvils (Vickers Hardness Number = 38). The load at fracture, measured in newton (N), is defined as the crushing strength. Each particle was tested at a new location between the anvils. Considering the small size of a particle, 30 particles could be tested on a surface of 20×20 mm. The average crushing strength of 30 or more of the particles is taken as the crushing strength of the batch. Weibull statistics were also used to determine the characteristic crushing

strengths of these batches, which were between 5% and 10% higher than the averages. For the purpose of this study, the differences between batches were so significant that the average crushing strengths were adequate to compare different batches.

III.C. Annealing

In the fuel production process, particles are consolidated into a fuel element, such as a sphere, during annealing. However, for the experiments described here, graphite spheres into which holes had been drilled were used. The loose particles were placed in these holes. These spheres together with other spheres were heat treated in the annealing furnace. Heating, annealing (1950°C for 1 h), and cooling were carried out under vacuum.

III.D. Burn-Leach Method

The burn-leach test gives an indication of the integrity of the SiC layer. A representative sample is split from the batch of coated particles. To remove available carbon from the particles, the sample is heated to 800°C in air for 16 h under natural convection. The sample is then leached with concentrated boiling nitric acid for another 16 h. Those particles with some form of defect may leach some or all of the uranium from that particle. The uranium in the leaching solution is determined, and the result is expressed as the amount of uranium (mg) relative to the total uranium (kg) in the sample. This is defined as the uncontained uranium (mgU/kgU).

IV. RESULTS AND DISCUSSION

IV.A. Influence of IPyC, SiC, and OPyC Layers on the Crushing Strength of Standard Quality Coated Particles—Before and After Annealing

Table I shows the crushing strength of five standard quality batches from the ACF. These samples were taken after only the buffer (88 μm, 1.0 g/cm^3) and the IPyC (36 μm, 1.92 g/cm^3) layers had been deposited. The standard quality batches were all manufactured with exactly the same coating parameters. The average and standard deviation of the averages is indicated in the last two columns. This shows that the method is quite consistent considering that we are testing the crushing strengths of inherently brittle ceramic layers.

Table II shows the crushing strength from the same five standard quality batches from the ACF after the buffer, IPyC, and SiC (36 μm, 3.20 g/cm^3) layers have been deposited.

Table III compares the crushing strength of a standard batch with the addition of the consecutive layers and the influence that annealing has on these samples. Because of the yielding nature of the soft metal anvils, finite

TABLE I

IPyC Samples from Different Standard Quality Batches

Batch	H101	H103	H105	H107	H109	Average of Five Batches (N)	Standard Deviation (N)
Average crushing strength (N)	49.6	50.0	54.9	51.5	47.3	50.7	2.8
Standard deviation (N)	13.9	14.4	14.5	14.6	16.8	—	—

TABLE II

SiC Samples from Different Standard Quality Batches

Batch	H101	H103	H105	H107	H109	Average of Five Batches (N)	Standard Deviation (N)
Average crushing strength (N)	66.2	70.2	72.6	65.5	68.3	68.5	2.9
Standard deviation (N)	12.6	12.2	13.6	11.9	11.3	—	—

TABLE III

Influence of Annealing on the Crushing Strength for Standard Quality Batch H109 from the ACF

Treatment	IPyC Sample	SiC Sample	TRISO Sample
Before annealing			
Average (N)	47.3	68.3	122.2
Standard deviation (N)	16.8	11.3	11.2
After annealing			
Average (N)	31.6		86.4
Standard deviation (N)	13.5	—	12.4
Fraction of original crushing strength	0.67	—	0.71

element analysis[6] indicated that fracturing does not initiate at the contact but rather in the layer material between the anvils. This negates the effect of the contact between the anvils and the different types of layers. This is different from previous tests where hard metal anvils were employed. The crushing strength after the addition of a SiC layer is noteworthy. The crushing strength of the particles is now mainly that of the SiC layer with only a minor contribution due to that of the IPyC layer. This is due to the fact that the elastic modulus of the carbon layers is only ~5% that of SiC. Because of the bonding between the SiC and carbon layers, the strain in all the layers will be approximately the same strain. The stress in the SiC layer will therefore be much higher than that in the IPyC layer. The particle during compression will therefore fracture when the fracture stress in the SiC layer is exceeded, also precipitating fracture of the IPyC layer.

Table III shows that the addition of an OPyC layer increases the crushing strength from 68 N (SiC sample) to 122 N (TRISO sample). In the case of the SiC sample, the SiC is directly in contact with the anvil while the TRISO particle has a layer of carbon, which is much more pliable, between the SiC layer and the anvil. This contact effect is probably small as the particles, during testing, fail because of the tensile stress where it is not in contact with the anvil material. The diameter of the particle increases from 840 to 910 μm when the OPyC layer is added. The dimensions of the SiC layer, however, stay the same. Adding another carbon layer, with low elastic modulus, should not result in an appreciable increase in the crushing strength of the sample unless it contributes to the residual strength or substantially alters the imposed stress.

Table III also indicates that the crushing strengths after annealing of both the IPyC sample and the TRISO

52

sample deteriorated by ~30%. Annealing at 1950°C will reduce any residual stresses produced by the coating process. The reduction in crushing strength after annealing is probably due to the presence of compressive residual stresses that were relieved by the annealing heat treatment. The stress applied to a particle during testing will be the sum of the applied stress and residual stress. Any initial compressive stress during testing will first have to be eliminated before a particle can fracture. In practice during the manufacture of the fuel for the PBMR, particles will always be subjected to the annealing temperature used. The presence of residual stress due to coating will not contribute to the effective crushing strength during operation. The crushing strength of the standard batch after annealing (86 N) may be used to estimate the fracture strength of the SiC layer. Using the method[6] that was presented before, a value for the fracture strength was estimated to be 1400 MPa for the standard quality SiC. This is higher than most other values in literature.[12] Direct comparison is inappropriate because of the fundamental differences between this method and other methods[2–5] that were previously used. The use of hard anvils results in a small volume of highly stressed material in the vicinity of the contact. When the appropriate soft metal anvils are used, the crack initiates because of tensile stress that develops in a large volume of the layer material between the anvils.

IV.B. The Influence of SiC Coating Parameters on the Crushing Strength of Particles— Before and After Annealing

Table IV shows the coating parameters of six experimental batches of coated particles. During this series the deposition of the carbon layers was kept constant. Batches J01 to J06 were produced in the RCF. Mixtures of H_2, Ar, methyl trichloro silane (MTS), and propylene were used to produce SiC layers. The coating temperatures were lower than for the standard quality TRISO particles. The standard quality batch from the ACF (H109) is included for comparison with the experimental batches from the RCF. H109 was deposited using only H_2 and MTS.

Linear elastic analysis shows that to a first approximation the strength of the particle is directly related to the thickness of the SiC layer. This is due to the minor contribution of the inner and outer carbon layers to the crushing strength. The deposition times were unfortunately not yet optimized to produce a SiC thickness of ~36 μm. To compensate for this, the crushing strengths were normalized to 36 μm by adjusting the crushing strength in direct linear relation to the thickness. Table V shows the influence of gas composition on the crushing strength of particles before and after annealing.

Batches J03 and J05 initially appeared to be promising as they had higher crushing strengths than the standard quality batch. The SiC density was also high enough to be within specification for a TRISO particle. However, annealing caused a drastic deterioration in the crushing strength of these batches. It was previously reported[13] that the addition of 80% Ar to the gas mixture resulted in an increase in the size of SiC crystals and an increase in the excess carbon that is included in the SiC layer. The crystal size and deviation from stoichiometry in the SiC layer could be the reasons for the significant difference in the behavior of these batches compared to the standard quality batch (Table III). Varying coating conditions may, through deviations in stoichiometry, introduce residual stresses in the SiC layer that vary from batch to batch. Although these batches have SiC layers that are within the specified density range and very hard SiC layers are formed,[13] the crushing strength of these particles after annealing may disqualify them from use in a reactor.

The increase in crystal size and excess carbon is also present when the SiC deposition temperature is increased. A lower flow rate of Ar is required to fluidize the particles than in the case with H_2. Also, the specific heat capacity of H_2 is higher than that of Ar. These two factors may result in the surface temperature of the particles in the reaction zone being higher when Ar is used as a carrier gas. The two photographs in Fig. 2 illustrate this

TABLE IV

SiC Deposition Parameters

Batch	Ar (%)	MTS (%)	Propylene (%)	Deposition Temperature (°C)	SiC Density (g/cm³)	SiC Thickness (μm)
J01	50	5.0	0.25	1300	3.175	45
J02	20	5.4	0.22	1300	3.146	42
J03	80	5.9	0.28	1300	3.189	41
J04	20	4.8	0.22	1300	3.118	40
J05	80	4.7	0.28	1300	3.189	34
J06	20	3.0	0.17	1400	3.173	54
H109 from ACF	0	1.2	0	1510	3.20	36

TABLE V

Influence of Coating Parameters on the Crushing Strength of Particles Before and After Annealing

Batch	Argon/MTS/PP (%)	Before Annealing[a] (N)	After Annealing[a] (N)	Fraction of Original Crushing Strength
J01	50/5.0/0.25	77	45	0.58
J02	20/5.4/0.22	111	88	0.80
J03	80/5.9/0.28	134	61	0.45
J04	20/4.8/0.22	104	101	0.97
J05	80/4.7/0.28	133	58	0.44
J06	20/3.0/0.17	79	67	0.84
H109	0/1.2/0.00	122	86	0.70

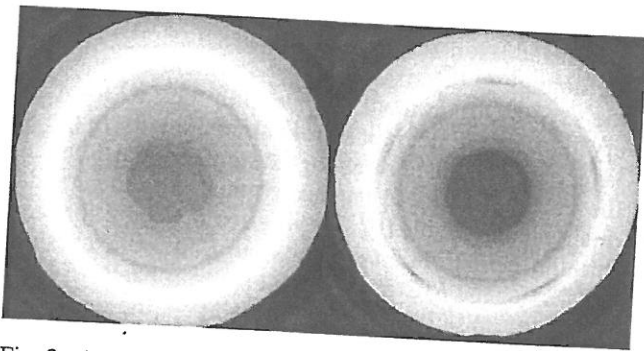[a]Linearly normalized to correspond to a SiC thickness of 36 μm.

Fig. 2. Appearance from top of empty RCF using Ar (left) and H$_2$ (right).

effect. These photographs were taken through the top sight-glass at the RCF while it was empty. The temperature of the coater tube was at 1050°C. At higher temperatures, which are used for coating SiC, the effect is more pronounced but difficult to photograph. It is clear that the temperature of the gas distributor at the bottom (in the center of each photo) and the surrounding area is lower (darker) when H$_2$ is used. At the time, a distributor with five inlet nozzles was used. The photograph on the right also shows the areas where each H$_2$ jet impinges on the side of the coater. These can be seen as five distinctly darker lines in the brightest circle around the gas distributor. Using argon in the fluidization mixture increases the temperature in the reaction zone, which may be the cause of the larger crystals and excess carbon in the SiC layer.

Before annealing, batch J04 had a lower crushing strength (95% confidence) than batch H109 (the standard quality batch). However, after annealing with more than a 99% confidence limit, the crushing strength of batch J04 was higher than the standard quality batch. The crushing strength of batch J04 did not decrease with annealing.

Under these coating conditions, however, excess Si may be introduced into the SiC layer, which also may disqualify it for use in a reactor. Again the change in stoichiometry of the SiC layer may affect the crushing strength before and after annealing.

From Table V it is clear that the reduction in crushing strength before and after annealing varies significantly from batch to batch. The most likely causes of this variation are the deviation from stoichiometry in the SiC layer and the differences in the residual stress produced during coating. The fraction of the original crushing strength is indicated in Table V and can be a useful indicator when determining the optimum coating conditions for the SiC layer.

The reduction in the crushing strength of annealed TRISO batches confirms the observations made in Ref. 2. In the current study the crushing strength of the standard TRISO batch reduced from 122 N to 86 N. In the previous study in Ref. 2, the average of the batches decreased from 21 N to 18 N. The lower measured crushing strengths[2] were probably due to the hard anvils that were used, as indicated in Ref. 6. Different coater configurations[2] that were used also resulted in a large spread in the results. The crushing strengths after annealing[2] were between 0.5 and 0.94 of the crushing strength before annealing, compared to the 0.7 measured for the standard quality batch in this study. In the study in Ref. 2 it was also found that the kernel influences the crushing strengths of batches. In other studies[6,7] using zirconia kernels, annealing resulted in an increase in crushing strength. The increase in crushing strength in this instance can be due to coating conditions that resulted in tensile residual stresses that were relieved during annealing. Furthermore, the density of the IPyC and OPyC layers in these batches[6,7] was ~1.6 to 1.7 g/cm^3, which is below the required densities for standard quality batches. The densities of the IPyC and OPyC layers that were used in this study varied between 1.92 and 2.0 g/cm^3.
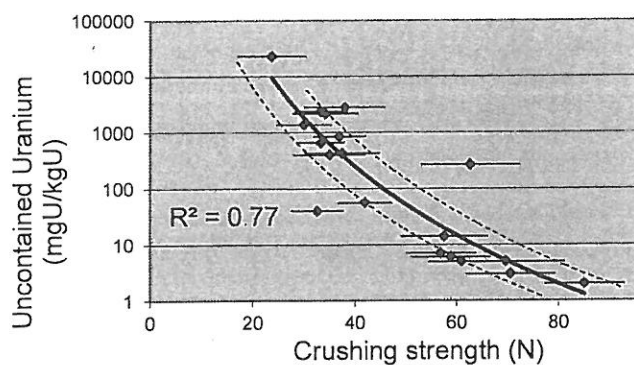
Fig. 3. Relationship between the crushing strength and the defective particles.

IV.C. Correlation Between Crushing Strength of Particles and the Burn-Leach Result

In the past, excessive high burn-leach results were ascribed to mechanical damage that occurred during handling.[9–11] Particles with a weak crushing strength should be more vulnerable to damage than particles with a higher crushing strength. Particles with a weak crushing strength may have defects that will also show up in burn-leach results. Figure 3 shows that there is indeed such a correlation between the crushing strength and the burn-leach result of a batch. The dashed lines indicate the standard deviation. The uncontained uranium (burn-leach result) and the crushing strengths were compared among batches of particles having a similar SiC layer. The SiC layer thickness and density for all of the batches varied from 15 to 19 μm and 3.16 to 3.19 g/cm^3, respectively.

V. CONCLUSIONS

Batches of particles were prepared using the prescribed standard conditions as well as a variety of other gas mixtures and temperatures. The crushing strengths of the batches of particles were determined by crushing between soft anvils. It was possible to test the crushing strength at the various stages of the coating process and to assess the influence of annealing on the crushing strength.

The tests showed that there are significant differences in the crushing strength of the batches of particles that have been produced with different coating parameters. The influence of the Ar content when depositing the SiC layer can be ascribed to the reduction in the heat capacity of the gas mixture. This results in higher deposition temperatures on the surface of particles, larger SiC crystals, and deviation from stoichiometry of the SiC layer. At a depositing temperature of 1300°C, an increase

in the Ar content resulted in particles with a higher crushing strength. However, after annealing, the crushing strength of this batch deteriorated to less than half of its original crushing strength. The reduction in crushing strength due to annealing varied considerably depending on the coating parameters that were used. The reduction in crushing strength is possibly due to initial deviations in stoichiometry and the presence of residual stresses in the SiC layer

It is generally accepted that the burn-leach method detects only the coated particles physically damaged in the coating process or during handling. A batch of weak particles should be more vulnerable to damage due to physical impact than a batch with higher crushing strength. The available results indicated such an inverse correlation between the crushing strength and the burn-leach result of a batch.

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