ORNL/TM-2017/035

Acceptance Test Data for BWXT Coated Particle Batch 93164A

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February 2017

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OAK RIDGE NATIONAL LABORATORY
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ORNL/TM-2017/035

Fusion and Materials for Nuclear Systems Division

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Defective IPyC Fraction and Pyrocarbon Anisotropy

John D. Hunn
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ACRONYMS

2-MGEM	Two-Modulator Generalized Ellipsometry Microscope
AGR	Advanced Gas Reactor (Fuel Development and Qualification Program)
AGR-5/6/7	Fifth/sixth/seventh AGR program irradiation experiments
ATR	Advanced Test Reactor
BWXT	BWX Technologies
CVD	Chemical vapor deposition
DAM	Data Acquisition Method
DRF	Data Report Form
INL	Idaho National Laboratory
IPyC	Inner pyrolytic carbon (TRISO layer)
IRF	Inspection Report Form
MTS	Methyl-trichlorosilane
N	Diattenuation
OPTAF	Optical anisotropy factor [OPTAF=(1+N)/(1-N)]
OPyC	Outer pyrolytic carbon (TRISO layer)
ORNL	Oak Ridge National Laboratory
PIP	Product Inspection Plan
PyC	Pyrolytic carbon or pyrocarbon
QC	Quality control
SiC	Silicon carbide (TRISO layer)
TRISO	Tristructural-isotropic (coated particles)
UCO	Uranium carbide/uranium oxide mixture (fuel kernels)

ACKNOWLEDGMENTS

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1. INTRODUCTION

Coated particle fuel batch J52O-16-93164 was produced by Babcock and Wilcox Technologies (BWXT) for possible selection as fuel for the Advanced Gas Reactor Fuel Development and Qualification (AGR) Program's AGR-5/6/7 irradiation test in the Idaho National Laboratory (INL) Advanced Test Reactor (ATR), or may be used as demonstration production-scale coated particle fuel for other experiments. The tristructural-isotropic (TRISO) coatings were deposited in a 150-mm-diameter production-scale fluidized-bed chemical vapor deposition (CVD) furnace onto 425-µm-nominal-diameter spherical kernels from BWXT lot J52L-16-69316[1]. Each kernel contained a mixture of 15.5%-enriched uranium carbide and uranium oxide (UCO) and was coated with four consecutive CVD layers: a ~50% dense carbon buffer layer with 100-µm-nominal thickness, a dense inner pyrolytic carbon (IPyC) layer with 40-µm-nominal thickness, a silicon carbide (SiC) layer with 35-µm-nominal thickness, and a dense outer pyrolytic carbon (OPyC) layer with 40-µm-nominal thickness. The TRISO-coated particle batch was sieved to upgrade the particles by removing over-sized and under-sized material, and the upgraded batch was designated by appending the letter A to the end of the batch number (i.e., 93164A).

Two samples riffled from upgraded TRISO batch 93164A were shipped to the Oak Ridge National Laboratory (ORNL) for quality control (QC) acceptance testing and analysis. The AGR-5/6/7 Fuel Specification, SPC-1352 [Marshall 2016], provides the requirements necessary for acceptance of the fuel manufactured for the AGR-5/6/7 irradiation test. The kernel QC acceptance testing and most of the coated particle QC acceptance testing was performed at BWXT and is not contained in this report. Two specified TRISO particle properties were measured at ORNL: pyrolytic carbon (PyC) anisotropy and defective IPyC fraction. The procedures for the ORNL characterization and QC acceptance testing of the particles are outlined in the ORNL Product Inspection Plan for AGR-5/6/7 Coated Particles, AGR-CHAR-PIP-28 [Hunn 2016], which is consistent with the INL Statistical Sampling Plan for AGR-5/6/7 Fuel Materials, PLN-4352 [Lybeck 2016].

Additional analysis of the x-ray images obtained for determination of the defective IPyC fraction was performed to identify and quantify any other obvious microstructural anomalies in the TRISO-coated particle sample. This additional analysis is briefly summarized in this report for information-only and is provided in greater detail in a separate report [Helmreich et al. 2017].

[1] Note that succeeding coater batches produced for possible use in the AGR-5/6/7 irradiation test were fabricated from BWXT kernel lot J52R-16-69317.

2. DEFECTIVE IPYC

Particles with excessive IPyC permeability can allow the infiltration of HCl into the buffer region of a TRISO particle during the initial stages of SiC deposition. HCl is a byproduct of the SiC CVD process when using hydrogen (H_2) and methyl-trichlorosilane (MTS) precursors. This HCl can react with the kernel and disperse uranium into the surrounding buffer and IPyC layers, especially when particles are heated to 1800°C during the compact manufacturing process. Excessive uranium dispersion can be detected by x-ray radiography of the TRISO-coated particles. Visual standards for what constitutes excessive uranium dispersion are included in the AGR-5/6/7 Fuel Specification and particles that exhibit excessive uranium dispersion are counted as having a defective IPyC coating. The ORNL Data Acquisition Method (DAM-47), Counting of TRISO Particles with Excessive Uranium Dispersion Inside SiC [Hunn 2013], provides the detailed procedures and requirements for the analysis that was performed to determine the defect IPyC fraction.

The fraction of particles with defective IPyC was determined for two subsamples riffled from TRISO Batch 93164A, Sample NP-C1364, which was a 130-gram sample riffled by BWXT and shipped to ORNL for analysis. Riffling of Sample NP-C1364 was performed according to the sampling instructions in Product Inspection Plan PIP-28 to obtain subsamples for analysis. The combined number of particles with defective IPyC in these two subsamples is reported on Inspection Report Form IRF-28A (Figure 1) with a determination as to whether the particle batch satisfied the specified parameters for this property. Batch 93164A does not meet the AGR-5/6/7 Fuel Specification requirements for the maximum defective IPyC fraction.

Figure 2 through Figure 7 are copies of the data report forms generated as part of the completion of Product Inspection Form PIP-28. Figure 2 is the particle weight determination used to ensure that each defect IPyC subsample had sufficient particles to meet the two acceptance test stages called out in the Statistical Sampling Plan for AGR-5/6/7 Fuel Materials. The minimum particle number requirements for this two-stage sampling appear in the acceptance criteria column in IRF-28A (Figure 1). Figure 3 is a record of the conditions of the particle heat treatment procedure, AGR-CHAR-DAM-41 [Kercher 2010], performed to simulate compact heat treatment and induce detectable uranium dispersion in particles with defective IPyC. The AGR-5/6/7 Fuel Specification specifies heat treatment of the compacts for at least 1 h between 1650°C and 1800°C. Particles were heated with the furnace schedule used for the compacts produced for the previous three AGR irradiation experiments. The loose particles were heated in a bed of graphite powder to minimize stress from temperature gradients. Figure 4 and Figure 6 are the individual results of the defective IPyC analysis for the two subsamples; these Data Report Forms (DRF-47) document the number of particles tested in each subsample and the number of particles counted as having defective IPyC based on their exhibition of excessive uranium dispersion. Figure 5 and Figure 7 are the associated DRF-47 supplemental data forms for the two analyzed subsamples and document the number of particles that had other anomalies of interest visible in the single x-ray radiograph image acquired of each particle. The supplemental data forms also report the fraction of particles in each subsample that exhibited each anomaly and a 95%-confidence prediction of the maximum fraction in the TRISO particle batch, based on the observed number, the subsample size, and using binomial distribution statistics. Details about these anomalies and additional images acquired by high-resolution x-ray tomography to further characterize them, as well as the uranium dispersion in the particles with defective IPyC, are available in a separate summary report [Helmreich et al. 2017].

Inspection Report Form IRF-28A: AGR-5/6/7 Coated Particles

Procedure:	AGR-CHAR-PIP-28 Rev. 1
Coated particle sample ID:	NP-C1364
Coated particle sample description:	TRISO particles from BWXT coating batch J52O-16-93164A

Property	Measured Data # of particles	Specification INL SPC-1352	Acceptance Criteria	Acceptance Test Value	Pass or fail	Data Records
Defective IPyC coating fraction (fraction of total particles)	119944	$\leq 1.0 \times 10^{-4}$	≤2 with excessive U dispersion in ≥62956 particles or ≤6 with excessive U dispersion in ≥118422 particles	25	fail	DRF-47

Comments

See NP-C1364-C01_DRF47R0 and NP-C1364-D01_DRF47R0 for individual results of defective IPyC measurement and summary of other anomalies observed by x-ray radiography. Sample also failed Stage 1 testing with 9 particles with excessive U dispersion out of 64007.

QC Supervisor — Date: 1-27-17

QA Reviewer — Date: 1/30/17

Figure 1. Inspection report for defective IPyC.

Data Report Form DRF-22: Estimation of Average Particle Weight

Procedure:	AGR-CHAR-DAM-22 Rev. 1
Operator:	Grant Helmreich
Particle sample ID:	NP-C1364-B00
Particle sample description:	Particles from BWXT coating batch J52O-16-93164A
Filename:	\\mc-agr\AGR\ParticleWeight\W16102801_DRF22R1.xls

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Weight of particles (g):	0.1469	0.1440	0.1212	0.1067	0.1397
Number of particles:	149	149	123	108	142
Average weight/particle (g):	9.86E-04	9.66E-04	9.85E-04	9.88E-04	9.84E-04

Mean average weight/particle (g):	9.819E-04
Standard error in mean average weight/particle (g):	3.92E-06

Operator — Date: 10/28/16

Figure 2. Data report for average particle weight measured for subsample riffling.

Data Report Form DRF-41: Heat-treatment of Loose Particles Using a Graphite Furance

Procedure:	AGR-CHAR-DAM-41 Rev. 0
Operator:	Darren Skitt/John Hunn
DRF filename:	\\mc-agr\AGR\Furnaces\H16103101_DRF41R0.xls

Particle loading procedure	AGR-CHAR-PIP-28-R1				
Particle weight (g)	118.0566	Additional Material	Graphite	AM weight (g)	N/A
Details	J52O-16-93164A, AGR-5/6/7 TRISO pre-production batch.				

Furnace calibration due date	10/13/17

Thermal schedule				
Ramp 1	20	°C/min to	1800	°C
Dwell 1	1		hr	
Ramp 2	-20	°C/min to	700	°C
Dwell 2	0		hr	
Ramp 3	N/A	°C/min to	50	°C
Dwell 3	0		hr	
Ramp 4		°C/min to		°C
Dwell 4			hr	

Sample loading (top to bottom)			
Sample ID	NP-C1364-C01 (63.0067 g)	Crucible Marking	X
Sample ID	NP-C1364-D01 (55.0499 g)	Crucible Marking	Y
Sample ID		Crucible Marking	
Sample ID		Crucible Marking	
Sample ID		Crucible Marking	

3 vacuum / gas purges	1	2	3
Heat-treatment atmosphere	Vacuum		
Flow rate	N/A		

Comments

Optical pyrometer calibration due 5/31/17.

John Hunn — Operator

10-31-16 — Date

Figure 3. Data report for particle heat treatment to simulate compact heat treatment.

Data Report Form DRF-47: Counting of Particles with Excessive Uranium Dispersion Inside SiC

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	John Hunn/Grant Helmreich
Particle sample ID:	NP-C1364-C01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93164A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161101\NP-C1364-C01_DRF47R0.xlsm

Weight of particles in sample (g):	63.0067
Number of particles in sample:	64007
Average weight/particle (g):	9.8437E-04

Number of particles with excessive U dispersion:	9

Comments

Tape mounts T16110101 thru T16110118.

Operator | Date

Figure 4. Data report for defective IPyC analysis of subsample NP-C1364-C01.

Data Report Form DRF-47 Supplemental: Counting of Defects and Anomalies by Radiography

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	John Hunn/Grant Helmreich
Particle sample ID:	NP-C1364-C01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93164A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161101\NP-C1364-C01_DRF47R0.xlsm

Number of particles in sample:	64007
Number of radiographs analyzed:	18

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	9	1.41E-04	2.5E-04
White spots	26	4.06E-04	5.7E-04
Thin or low density SiC	4	6.25E-05	1.5E-04
Extra layers	0	0.00E+00	4.7E-05
Missing kernel	5	7.81E-05	1.7E-04
Kernel migration	0	0.00E+00	4.7E-05
Missing buffer	0	0.00E+00	4.7E-05
Particles with kernel anomalies	1222	1.91E-02	2.1E-02
Dimple or facet	*494*	*7.72E-03*	*8.4E-03*
Severe dimple or facet	*NA*	*NA*	*NA*
Notched kernel	*778*	*1.22E-02*	*1.3E-02*
Irregular kernel	*77*	*1.20E-03*	*1.5E-03*
Multi-kernel	*4*	*6.25E-05*	*1.5E-04*

Comments

Severe dimple or facet not categorized for this analysis.

Operator

11/18/16

Date

Figure 5. Summary of anomalies observed during defective IPyC analysis of subsample NP-C1364-C01.

Data Report Form DRF-47: Counting of Particles with Excessive Uranium Dispersion Inside SiC

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	John Hunn/Grant Helmreich
Particle sample ID:	NP-C1364-D01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93164A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161101\NP-C1364-D01_DRF47R0.xlsm

Weight of particles in sample (g):	55.0499
Number of particles in sample:	55937
Average weight/particle (g):	9.8414E-04

Number of particles with excessive U dispersion:	16

Comments

Tape mounts T16110119 thru T16110134.

Operator

11/18/16

Date

Figure 6. Data report for defective IPyC analysis of subsample NP-C1364-D01.

Data Report Form DRF-47 Supplemental: Counting of Defects and Anomalies by Radiography

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	John Hunn/Grant Helmreich
Particle sample ID:	NP-C1364-D01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93164A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161101\NP-C1364-D01_DRF47R0.xlsm

Number of particles in sample:	55937
Number of radiographs analyzed:	16

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	16	2.86E-04	4.4E-04
White spots	23	4.11E-04	5.9E-04
Thin or low density SiC	5	8.94E-05	1.9E-04
Extra layers	0	0.00E+00	5.4E-05
Missing kernel	0	0.00E+00	5.4E-05
Kernel migration	2	3.58E-05	1.2E-04
Missing buffer	1	1.79E-05	8.5E-05
Particles with kernel anomalies	1590	2.84E-02	3.0E-02
Dimple or facet	*641*	*1.15E-02*	*1.3E-02*
Severe dimple or facet	*NA*	*NA*	*NA*
Notched kernel	*1030*	*1.84E-02*	*2.0E-02*
Irregular kernel	*87*	*1.56E-03*	*1.9E-03*
Multi-kernel	*2*	*3.58E-05*	*1.2E-04*

Comments

Severe dimple or facet not categorized for this analysis.

Operator

11/18/16

Date

Figure 7. Summary of anomalies observed during defective IPyC analysis of subsample NP-C1364-D01.

3. PYROCARBON ANISOTROPY

Pyrocarbon anisotropy is a key parameter that can influence the radiation behavior of the IPyC and OPyC layers in TRISO-coated particle fuel. Excessive preferred orientation of the graphene planes within the pyrocarbon layers can lead to overall asymmetric shrinkage and fracture under irradiation. Because of the very large anisotropy for the reflection of light polarized parallel to the graphene planes versus light polarized perpendicular to the graphene planes, determination of the PyC optical anisotropy (OPTAF), defined as the ratio of the maximum to minimum reflectivity of polarized light, can be used as a relative measure of the preferred orientation of the graphene planes within the layer. The ORNL Two-Modulator Generalized Ellipsometry Microscope (2-MGEM) uses advanced ellipsometry techniques to measure the diattenuation (N) of a material, which is related the optical anisotropy by OPTAF = (1+N)/(1-N) [Jellison and Hunn 2008]. The ORNL Data Acquisition Method (DAM-18), Data Acquisition Method for Measurement of Pyrocarbon Anisotropy Using the Second Generation Two-Modulator Generalized Ellipsometry Microscope [Hunn and Jellison 2016], provides the detailed procedures and requirements for the analysis that was performed to determine the optical anisotropy of the IPyC and OPyC layers.

Average optical anisotropies of the IPyC and OPyC layers were measured on polished cross sections of 10 particles from TRISO Batch 93164A Sample NP-C1305, which was a 0.41-gram sample riffled by BWXT and shipped to ORNL in advance of TRISO Batch 93164A Sample NP-C1364. Anisotropy measurements were not repeated on a riffled subsample from TRISO Batch 93164A Sample NP-C1364 per Product Inspection Plan PIP-28 because the measurement had been satisfactorily completed on the earlier sample.

The average optical diattenuation values of the inner and outer pyrocarbon layers are reported on Inspection Report Form IRF-28B (Figure 8) with a determination as to whether the particle batch satisfied the specified parameters for this property. Batch 93164A meets the AGR-5/6/7 Fuel Specification requirements for the IPyC and OPyC diattenuation. The Data Report Forms in Figure 9 and Figure 10 show the average anisotropy data for each particle cross section in terms of both the diattenuation and the OPTAF. Note that the standard deviation in the measured anisotropy within each layer was greater than the standard deviation in the distribution of measured values for the ten-particle sample. This illustrates that even though there is significant localized variation in the PyC microstructure within each layer, the average PyC anisotropy is relatively consistent from particle to particle.

Inspection Report Form IRF-28B: AGR-5/6/7 Coated Particles

Procedure:	AGR-CHAR-PIP-28 Rev. 1
Coated particle sample ID:	NP-C1305
Coated particle sample description:	TRISO particles from BWXT coating batch J52O-16-93164A

Property	Measured Data			k or t value	Specification INL SPC-923	Acceptance Criteria	Acceptance Test Value	Pass or fail	Data Records
	Mean (x)	Std. Dev. (s)	# measured (n)						
IPyC diattenuation	0.0132	0.0008	10	1.833	mean ≤ 0.0170	B = x + ts/√n ≤ 0.0170	0.014	pass	DRF-18
				3.981	dispersion ≤0.01 ≥0.0242	D = x + ks < 0.0242	0.016	pass	
OPyC diattenuation	0.0086	0.0005	10	1.833	mean ≤ 0.0122	B = x + ts/√n ≤ 0.0122	0.009	pass	DRF-18
				3.981	dispersion ≤0.01 ≥0.0242	D = x + ks < 0.0242	0.011	pass	

Comments
See R16080301_DRF18R3 for full results of diattenuation (N) measurements. Mean OPTAF=(1+N)/(1-N) was 1.0265 (IPyC) and 1.0174 (OPyC).

QC Supervisor — Date: 1-25-17

QA Reviewer — Date: 1/25/17

Figure 8. Inspection report for pyrocarbon anisotropy.

Data Report Form DRF-18A: Measurement of Pyrocarbon Anisotropy using the 2-MGEM2 - IPyC

Procedure:	AGR-CHAR-DAM-18 Rev. 3
Operator:	Grant Helmreich
Mount ID:	M16080201
Sample ID:	NP-C1305
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93164A
Folder containing data:	\\mc-agr\AGR\2-MGEM\R16080301\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0140	0.0032	0.0017	1.0284	0.0066	0.0035
2	0.0130	0.0034	0.0017	1.0263	0.0070	0.0035
3	0.0140	0.0034	0.0017	1.0284	0.0070	0.0035
4	0.0118	0.0033	0.0016	1.0239	0.0068	0.0033
5	0.0133	0.0028	0.0016	1.0270	0.0058	0.0033
6	0.0141	0.0033	0.0017	1.0286	0.0068	0.0035
7	0.0132	0.0028	0.0016	1.0268	0.0058	0.0033
8	0.0124	0.0029	0.0016	1.0251	0.0059	0.0033
9	0.0126	0.0032	0.0016	1.0241	0.0061	0.0033
10	0.0132	0.0036	0.0016	1.0268	0.0074	0.0033
Average	0.0132	0.0032	0.0016	1.0265	0.0065	0.0034
St. Dev.	0.0008	0.0003	0.0001	0.0017	0.0006	0.0001

Comments
Sample NP-C1305 was a 0.41 gram sample riffled by BWXT and shipped to ORNL in July 2016. Because the sample was already an appropriately-small size, no riffling was done at ORNL and particles were selected for analysis directly from the received sample.

Operator

Date

Figure 9. Data report for IPyC anisotropy.

Data Report Form DRF-18B: Measurement of Pyrocarbon Anisotropy using the 2-MGEM2 - OPyC

Procedure:	AGR-CHAR-DAM-18 Rev. 3
Operator:	Grant Helmreich
Mount ID:	M16080201
Sample ID:	NP-C1305
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93164A
Folder containing data:	\\mc-agr\AGR\2-MGEM\R16080301\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0092	0.0034	0.0019	1.0186	0.0069	0.0039
2	0.0077	0.0027	0.0017	1.0155	0.0055	0.0035
3	0.0088	0.0033	0.0018	1.0178	0.0067	0.0037
4	0.0087	0.0033	0.0017	1.0176	0.0067	0.0035
5	0.0082	0.0030	0.0017	1.0165	0.0061	0.0035
6	0.0091	0.0033	0.0018	1.0184	0.0067	0.0037
7	0.0081	0.0028	0.0017	1.0163	0.0057	0.0035
8	0.0086	0.0028	0.0017	1.0173	0.0057	0.0035
9	0.0092	0.0032	0.0016	1.0186	0.0065	0.0033
10	0.0087	0.0031	0.0017	1.0176	0.0063	0.0035
Average	0.0086	0.0031	0.0017	1.0174	0.0063	0.0035
St. Dev.	0.0005	0.0003	0.0001	0.0010	0.0005	0.0002

Comments
Sample NP-C1305 was a 0.41 gram sample riffled by BWXT and shipped to ORNL in July 2016. Because the sample was already an appropriately-small size, no riffling was done at ORNL and particles were selected for analysis directly from the received sample.

Operator

8/5/16

Date

Figure 10. Data report for OPyC anisotropy.

4. CONCLUSION

The analyses called out in the ORNL Product Inspection Plan for AGR-5/6/7 Coated Particles were completed as part of the acceptance testing of BWXT TRISO-coated particle Batch 93164A. Subsamples were analyzed by x-ray radiography to look for the uranium dispersion that is a marker for defective IPyC layers and with the ORNL 2-MGEM to measure the optical anisotropy of the pyrocarbon layers. The AGR-5/6/7 Fuel Specification requirements for IPyC and OPyC anisotropy were satisfied but the maximum defective IPyC fraction was above the specified limit of $\leq 10^{-4}$. X-ray radiography identified 25 particles with excessive uranium dispersion out of the 119944 particles analyzed. This gives a measured defective IPyC fraction of 2.08×10^{-4} in the material tested and a 95%-confidence prediction of the maximum fraction in the TRISO particle batch of 2.92×10^{-4}.

5. REFERENCES

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