TGA Study for Fibres Desizing

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6 June 2024

Motivation of the Study

- Fibre insulation on Nb_3Sn cables contains **sizing**, which is important for braiding.
- During the coil reaction, this sizing decomposes into a grey, **conductive residue**.
- The coil reaction is in an **argon atmosphere** (no oxygen), so the sizing cannot fully burn off.

Without sizing, fibres undergo filamentation and breakage.

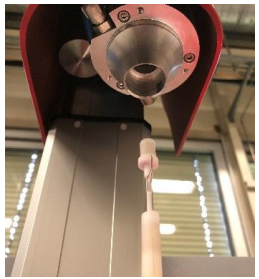
Nb_3Sn cable insulation before and after coil reaction.

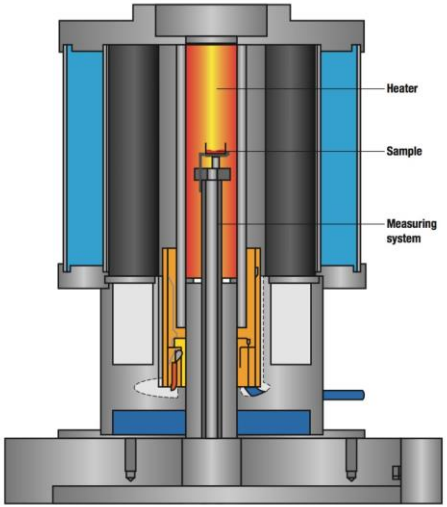
Questions

1. Can the sizing be removed with a thermal cycle in oxygen?
2. What temperature does the sizing burn?
3. How long does it take to burn it? – Cable conductor oxidation
4. Is it easier to remove sizing after the coil reaction?

Thermogravimmetric Analysis (TGA)

- A known mass of sample is heated with a fixed ramp rate in a controlled atmosphere and the loss of mass is measured with high precision .
- The presence of sizing is associated with a loss in mass.





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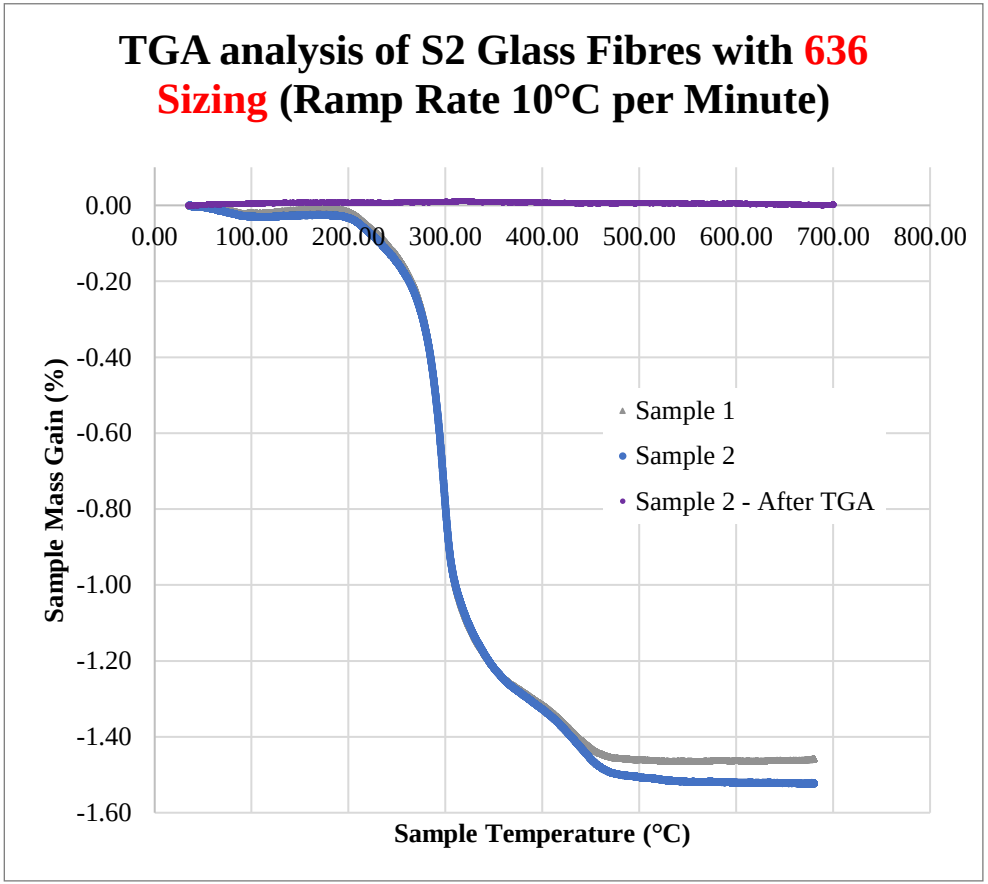
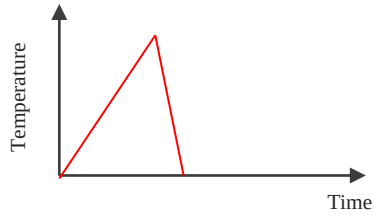


Planned Measurements

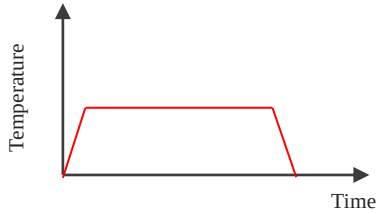
Dynamic - Heat to 700°C



Allows to determine **onset temperature** of sizing burning.

- 636 (starch*)
- 493 (epoxy resin*)
- 933 (silane*)
- QS1318 - Quartzel (silane*)

Static - Heat to temperature and hold.



Allows to determine **time taken** for desizing.

- 636
- QS1318

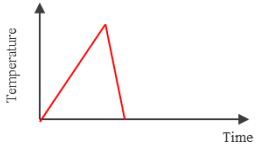
Repeat on reacted fibres

Properties of S2 Glass and Quartzel Fibres

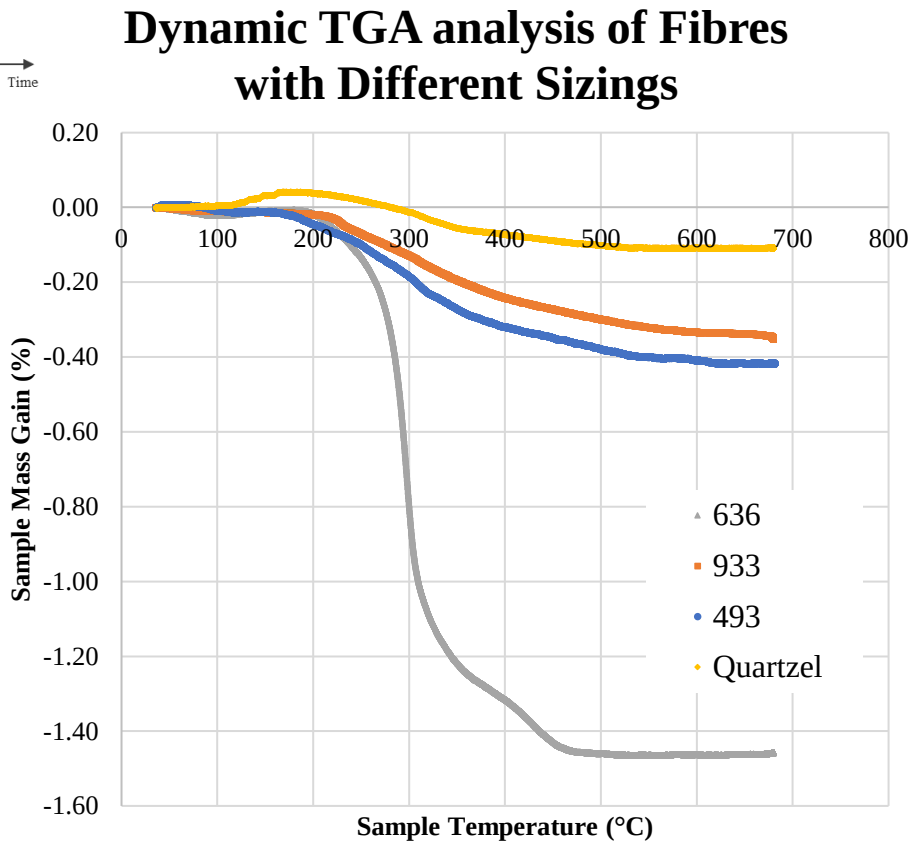
Property	Unit	S2 Glass	Quartzel
Softening Point	°C	1056	1700
Annealing Point	°C	816	1220
Strain Point	°C	766	1050 (deduced from datasheet)

*main component of sizing

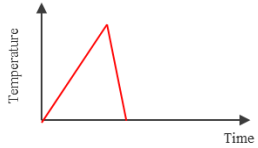
Initial Results
(Sizings Comparison)



- Samples with different sizings were heated to 700°C in air, and cooled.
- Important differences in mass loss between sizings.
  - Quartzel fabric appears to have less sizing, which could explain previously observed differences in electrical properties.
- Onset temperature of decomposition appears to be at 200°C.
- Mass of Quartzel goes up?



Initial Results
(Reacted vs. As-Received)

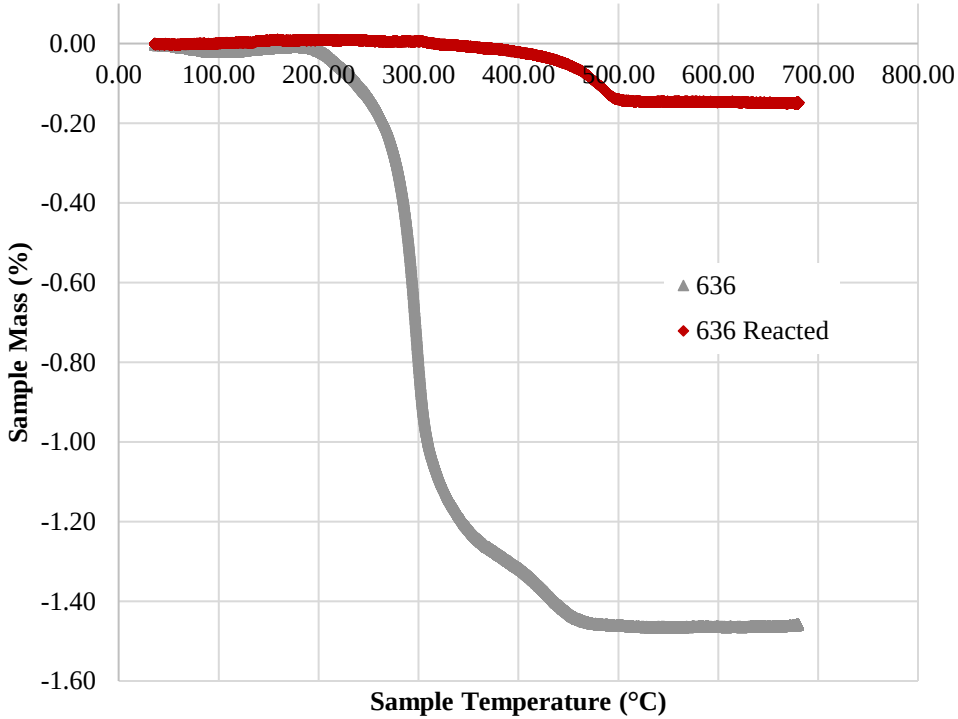


- The mass loss was significantly less for reacted fibres than for as-received ones.
  - A large mass of sizing may be removed during the Nb_3Sn reaction cycle in argon.
- The carbon residue appears to begin combusting at higher temperatures (400 °C).

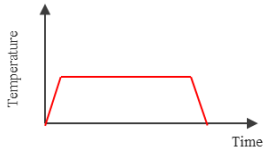
Apprearance of As-Received Fibres

Appearance of Reacted Fibres

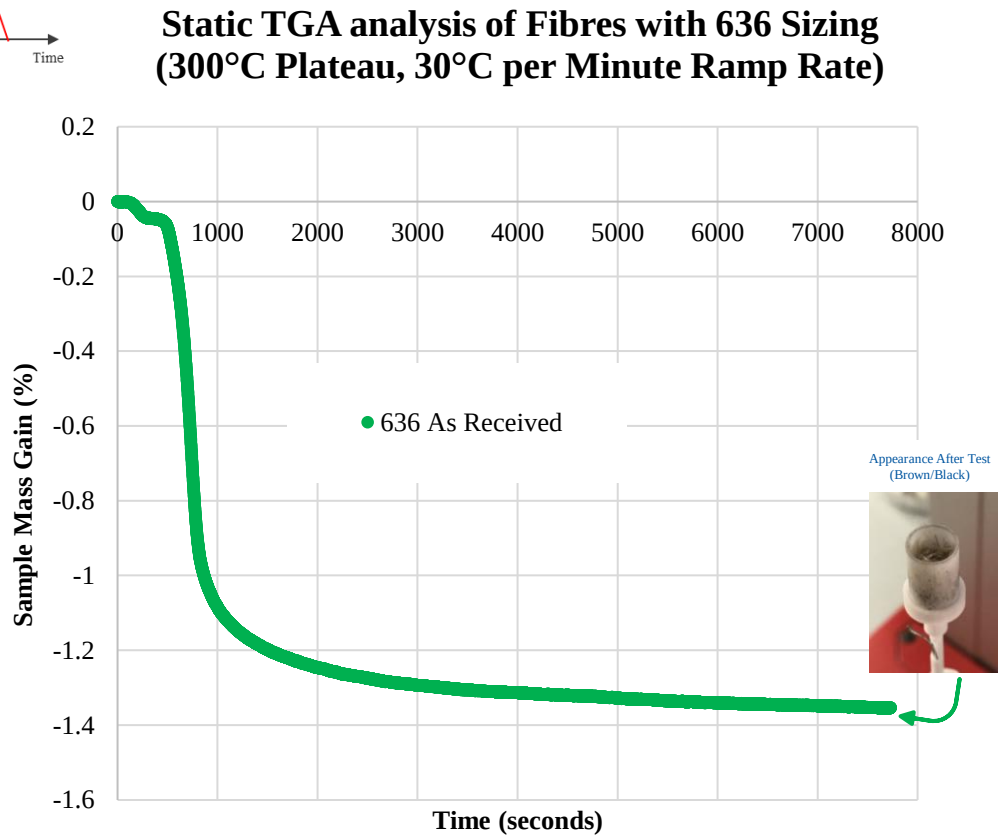
Dynamic TGA analysis of Fibres with 636 Sizing As Received vs. Reacted (Ramp Rate 10°C per Minute)



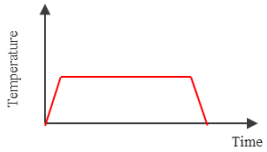
Initial Results
(300°C Plateau)



- Fibres with 636 sizing were heated to 300°C and kept for 2 hours in air.
- Significant mass loss after ~30 minutes, but not all the sizing was removed.
- Evident from:
  1. The **colour of the fibres (brown/black)**
  2. The sample not reaching the **expected mass loss (~1.5%).**
- There must be a component that burns at higher temperatures.



Initial Results
(300°C Plateau)

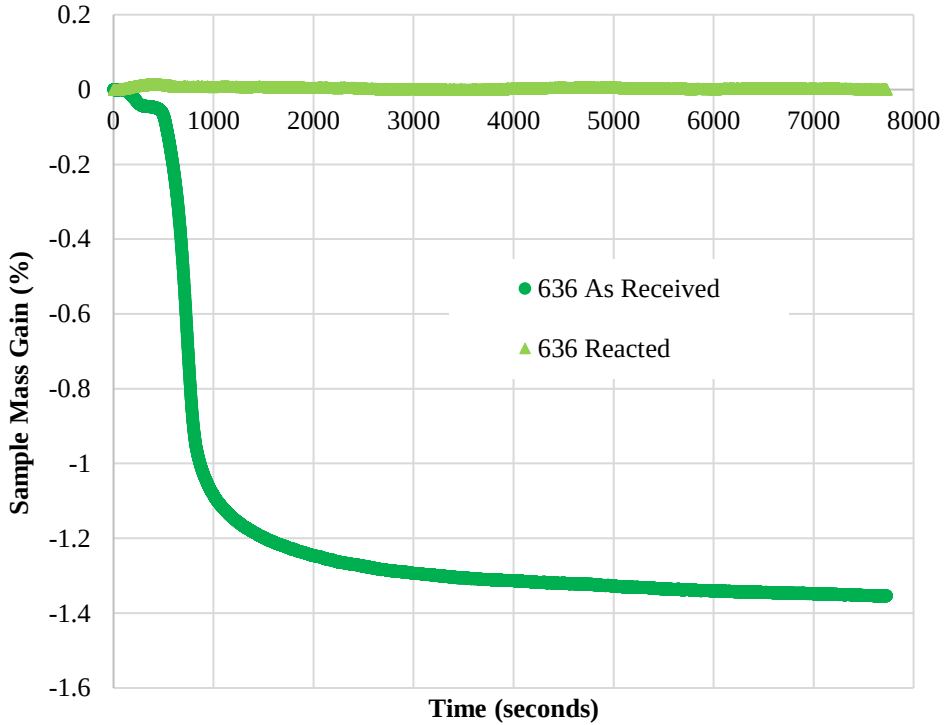


- The same test was performed on fibres with 636 sizing which had undergone a **Nb_3Sn reaction cycle** in argon.
- There was **negligible mass loss** for these samples.
- This suggests that the **carbon residue on fibres does not burn at 300°C.**

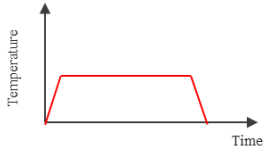
Apprearance of As-Received Fibres Before Analysis

Appearance of Reacted Fibres Before Analysis

Static TGA analysis of Fibres with 636 Sizing (300°C Plateau, 30°C per Minute Ramp Rate)



Initial Results
(400°C Plateau)

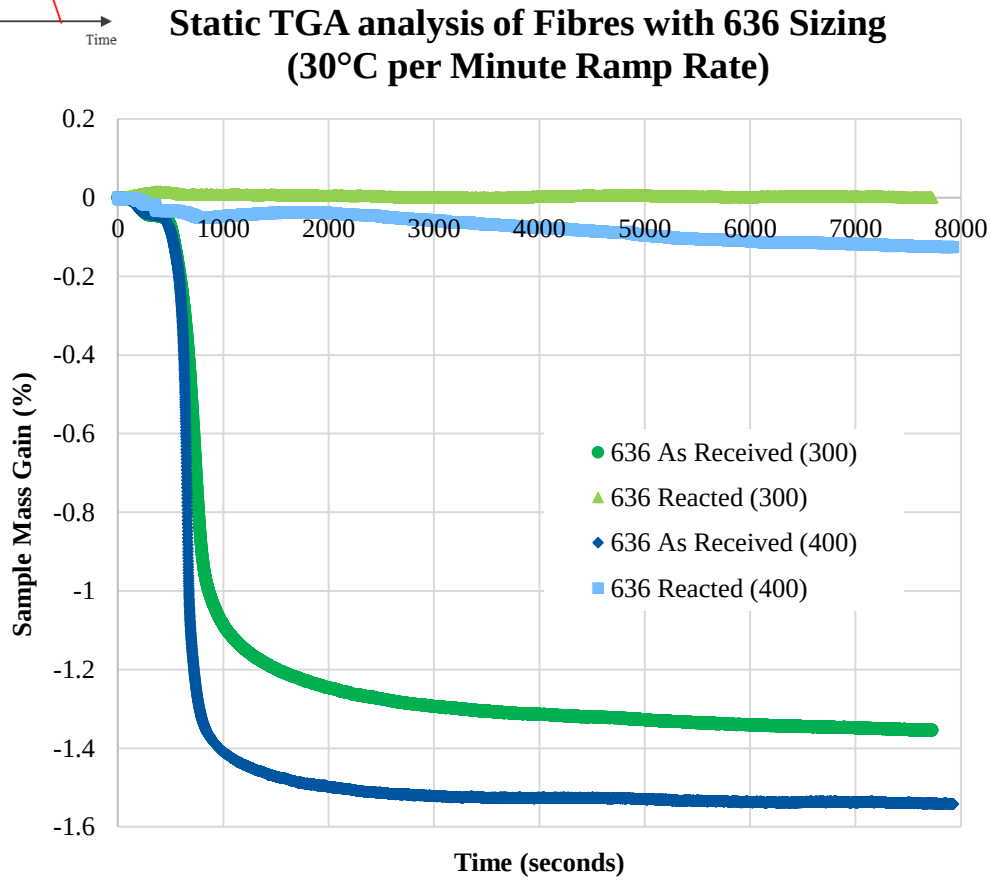


- Heating the fibres to 400°C appears to be more effective at desizing the fibres than 300°C.
- A greater mass loss is observed compared to 300°C, and the fibres generally have a cleaner appearance.
- In the reacted fibres, the sizing is removed more gradually.

As-Received Fibres after **300°C** Static TGA

As-Received Fibres after **400°C** Static TGA

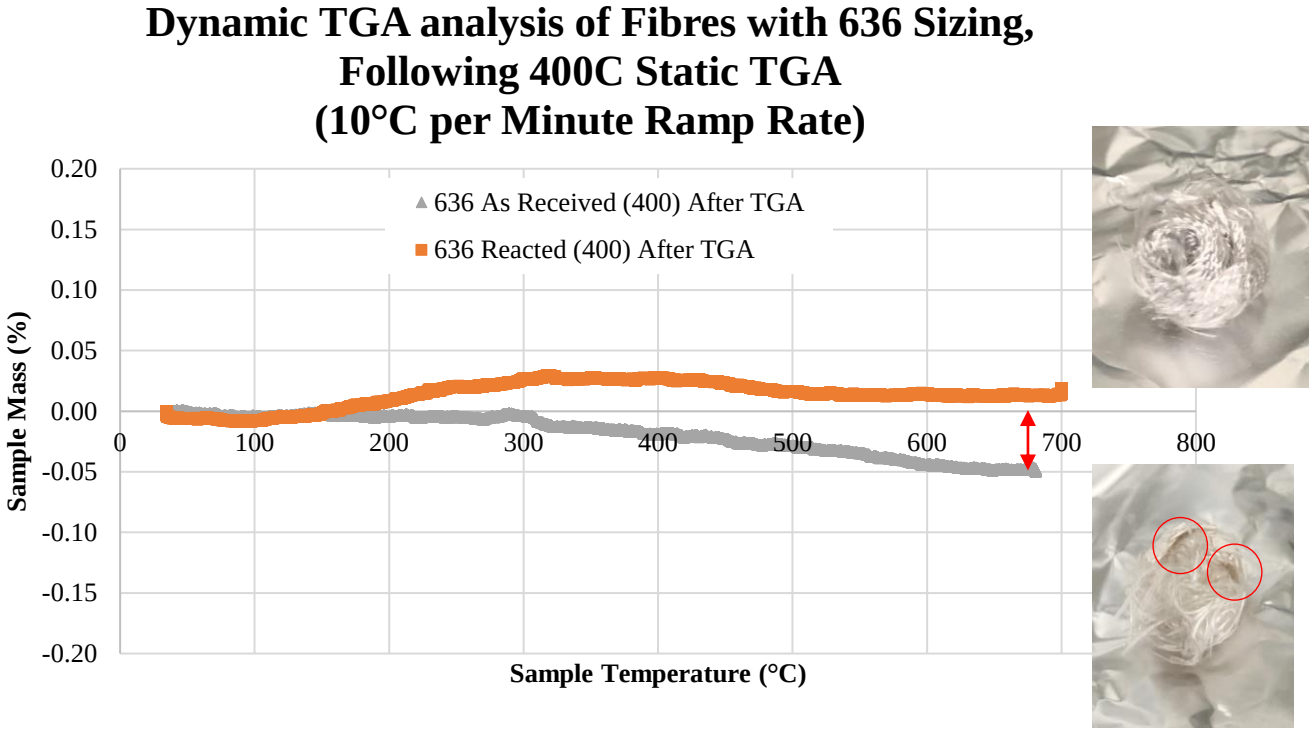
Reacted Fibres after **400°C** Static TGA



Possible Residual Sizing after 400°C Static TGA

- Dynamic TGA was performed on the samples which received the 400°C Static TGA, to confirm if the sizing was fully removed.
- There was a small, but measurable mass loss for the as-received fibres.
- This could be from the brown patches visible on the fibres (shown in red circles).

400°C Static TGA → 700°C Dynamic TGA



Summary

1. There are differences in mass loss between sizings.
   - (636 > 493 > 933 > QS1318)

1. The sizings all begin losing mass at around 200°C.

2. 636 sizing is only partially removed with a 300°C cycle.
   - There is likely a component with a higher decomposition temperature.

3. A 400°C cycle appears to be more effective at removing sizing.

4. The conductive residue from 636 sizing was also removed at 400 °C, however it required approximately 2 hours to burn off.

Conclusions & Further Work

- The amount of sizing (mass percent) is different for the various fibres.
  - This is likely due to the manufacturer’s specifications.
- Desizing should be performed at temperatures greater than 300°C, for fibres with 636 sizing.
- 400°C is a good starting point, because it also works for reacted fibres.
- Tests to be performed on Quartzel fibres, reacted and not reacted. With starch sizing.

Additional Slides

PRODUCT INFORMATION

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636 S-2 Glass® Yarn

High-Strength Solutions for Your Toughest Reinforcement Challenges

AGY's S-2 Glass® high-strength fibers are specifically designed to meet your most demanding performance processing and cost requirements. AGY's global network of people and facilities are ready to help you develop innovative solutions to your most difficult reinforcement challenges.

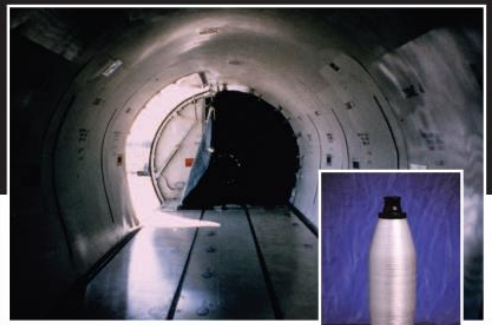
Product Application
636 S-2 Glass yarn is designed to be used in transportation, construction and recreation applications such as:
- Aircraft cargo liners
- High temperature insulation
- High temperature filtration
- Surfboards

Product Solutions
S-2 Glass fibers offer a unique combination of properties: strength, impact resistance, stiffness, radar transparency and temperature and fatigue resistance. Compared with other reinforcing materials, S-2 Glass fibers weigh less than conventional glass fiber and deliver better cost performance than aramid and carbon fibers. In addition, these yarns meet the requirements of MIL-Y-114OH specifications.

Product Description
636 S-2 Glass direct sized yarns consist of numerous filaments of varying diamters, twisted to form yarns. The material's starch-oil sizing, which protects the glass filaments from abrasion during processing, is typically removed after processing and the fabric is treated with a resin compatible finish.

Resin Compatibility
- Starch-oil sized for weaving and processing

Processes
- Weaving
- Braiding
- Plying



Aircraft Cargo Liner

S-2 Glass Fiber Yarn

Features	Benefits
S-2 Glass fiber offers significantly more strength than conventional glass fiber: 85% more tensile strength in resin impregnated strands	Consistent high performance for reliable and durable finished parts
Better fiber toughness, modulus of resilience and impact deformation than conventional glass fiber	Improved impact capabilities to finished parts and higher composites durability and damaged tolerance
Softening point: 1056°C (1932°F) Annealing point: 816°C (1500°F) Strain point: 766°C (1410°F)	Greater fiber tensile strength and stability at elevated temperatures in thermoset and thermoplastic applications
Enhanced stiffness	Delivers 25% more linear-elastic stiffness than conventional glass fiber
Excellent tolerance to damage accumulation	The ability of composite parts to withstand high levels of tension and flexural fatigue without catastrophic failure
S-2 Glass fibers deliver 20% reduction in dielectric constant over E-Glass fibers	Radar transparency
Long shelf life, good machinability and excellent durability	Consistent performance and reliability
S-2 Glass fiber is an organic material	Fire resistant
S-2 Glass fiber's pristine white color provides consistent cosmetics	Consistent appearance

PRODUCT INFORMATION

Available Products

Yarn Type (metric)	Construction	Nominal Twist TPM	Nominal Twist TPI	Sizing	Approximate Yarn Diameter mm	Approximate Yarn Diameter inch	Nominal Bareglass Yield TEX	Nominal Bareglass Yield Yard/Pound	Denier	Nominal Filament Diameter
SCG75 (SC9 66)	1/0	Z40	1.0Z	636	0.192	0.0076	66	7,500	594	"G" or 9 microns
SCG150 (SC9 33)	1/0	Z40	1.0Z	636	0.136	0.0054	33	15,000	297	"G" or 9 microns
SCD450 (SC5 11)	1/0	Z40	1.0Z	636	0.076	0.0030	11	45,000	99	"D" or 5 microns

Glass Composition
"S Glass" - reference ASTM C 162-98, ISO 2078, MIL-S-13949H

Solids (% LOI*)
0.670 minimum
1.17 nominal
2.17 maximum
* Loss on ignition after drying

Additional References
Customer acceptance standard: TP-378

Packaging

Package #	7636	
Type Build	Double Taper	
Descriptions	Metric (cm)	English (in)
Inside diameter	6.05	2.38
Tube length	35.6	14.0
Traverse	30.9	12.18
Flange diameter	10.1	3.96
Maximum full package diameter	11.4	4.5
Minimum package weight	0.23kg	0.50lbs
Packages/carton	54 (18x3 layers)	
Cartons/pallet	4	
Packages/pallet	216	
Approximate net weight/pallet	500kg	1100lbs
Pallets/typical truckload	40	

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PRODUCT INFORMATION

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493 S-2 Glass® Yarn

High-Strength Solutions for Your Toughest Reinforcement Challenges

AGY's S-2 Glass® high-strength fibers are specifically designed to meet your most demanding performance processing and cost requirements. AGY's global network of people and facilities are ready to help you develop innovative solutions to your most difficult reinforcement challenges.

Product Application
493 S-2 Glass yarn is designed to be used for producing composite hybrids and structural laminates in the woven and braided markets. Common applications include:
- Wire insulation
- High performance sails
- Snowboards and skis

Product Solutions
S-2 Glass fibers offer a unique combination of properties: strength, impact resistance, stiffness, radar transparency and temperature and fatigue resistance. Compared with other reinforcing materials, S-2 Glass fibers weigh less than conventional glass fiber and deliver better cost performance than aramid and carbon fibers. In addition, these yarns meet the requirements of MIL-Y-114OH specifications.

Product Description
493 S-2 Glass direct sized yarns consist of numerous G-filament (9 micron) continuous glass strands, twisted and treated with a sizing compatible with epoxies or polyesters.

Resin Compatibility
- Epoxy
- Polyester

Processes
- Weaving
- Braiding

S-2 Glass Fiber Yarn

K-2 Skiis

Features	Benefits
S-2 Glass fiber offers significantly more strength than conventional glass fiber: 85% more tensile strength in resin impregnated strands	Consistent high performance for reliable and durable finished parts
Better fiber toughness, modulus of resilience and impact deformation than conventional glass fiber	Improved impact capabilities to finished parts and higher composites durability and damaged tolerance
Softening point: 1056°C (1932°F) Annealing point: 816°C (1500°F) Strain point: 766°C (1410°F)	Greater fiber tensile strength and stability at elevated temperatures in thermoset and thermoplastic applications
Enhanced stiffness	Delivers 25% more linear-elastic stiffness than conventional glass fiber
Excellent tolerance to damage accumulation	The ability of composite parts to withstand high levels of tension and flexural fatigue without catastrophic failure
S-2 Glass fibers deliver 20% reduction in dielectric constant over E-Glass fibers	Radar transparency
Long shelf life, good machinability and excellent durability	Consistent performance and reliability

PRODUCT INFORMATION

Available Products

Yarn Type	Construction	Nominal Twist TPM	Nominal Twist TPI	Sizing	Approximate Yarn Diameter mm	Approximate Yarn Diameter inch	Nominal Bareglass Yield TEX	Nominal Bareglass Yield Yard/Pound	Denier	Nominal Filament Diameter
SCG75 (SC9 66)	1/0	Z40	1.0Z	493	0.191	0.0076	66	7,500	594	"G" or 9 microns
SCG150 (SC9 33)	1/0	Z40	1.0Z	493	0.136	0.0054	33	15,000	297	"G" or 9 microns

Glass Composition
"S Glass" - reference AMS 3832A, ASTM C 162-90, ISO 2078

Solids (% LOI*)
0.20 minimum
0.45 nominal
1.29 maximum
* Loss on ignition after drying

Additional References
Customer acceptance standard: TP-378

Packaging

Package #	7636	
Type Build	Double Taper	
Descriptions	Metric (cm)	English (in)
Inside diameter	6.05	2.38
Tube length	35.6	14.0
Traverse	30.9	12.18
Flange diameter	10.1	3.96
Maximum full package diameter	11.4	4.5
Minimum package weight	0.23kg	0.50lbs
Maximum package weight	2.31kg	5.1lbs
Packages/carton	54 (18x3 layers)	
Cartons/pallet	4	
Packages/pallet	216	
Approximate net weight/pallet	500kg	1100lbs
Pallets/typical truckload	40	

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PRODUCT INFORMATION

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933 S-2 Glass® Yarn

High-Strength Solutions for Your Toughest Reinforcement Challenges

AGY's S-2 Glass® high-strength fibers are specifically designed to meet your most demanding performance processing and cost requirements. AGY's global network of people and facilities are ready to help you develop innovative solutions to your most difficult reinforcement challenges.

Product Application

933 S-2 Glass yarn is designed to be used in aerospace, defense and recreation applications such as:

- Radomes
- Leading and trailing edges of aircraft wings

Product Solutions

S-2 Glass fibers offer a unique combination of properties: strength, impact resistance, stiffness, radar transparency and temperature and fatigue resistance. Compared with other reinforcing materials, S-2 Glass fibers weigh less than conventional glass fiber and deliver better cost performance than aramid and carbon fibers. In addition, these yarns meet the requirements of MIL-Y-1140H specifications.

Product Description

933 S-2 Glass direct sized yarns consist of numerous G-filament (9 microns) continuous glass strands, twisted to form yarns and treated with a thermally stable inorganic sizing for high temperature matrices.

Resin Compatibility

- Polyamide • Phenolic • Polyimide
- Bis-Maleimides • Epoxy
- Polyetherimide • Polyetheretherketone
- Liquid Crystal Polymers
- Cyanate Ester

Processes

- Weaving • Braiding

Aircraft Radome

S-2 Glass Fiber Yarn

Features	Benefits
S-2 Glass fiber offers significantly more strength than conventional glass fiber: 85% more tensile strength in resin impregnated strands	Consistent high performance for reliable and durable finished parts
Better fiber toughness, modulus of resilience and impact deformation than conventional glass fiber	Improved impact capabilities to finished parts and higher composites durability and damaged tolerance
Softening point: 1056°C (1932°F) Annealing point: 816°C (1500°F) Strain point: 766°C (1410°F)	Greater fiber tensile strength and stability at elevated temperatures in thermoset and thermoplastic applications
Enhanced stiffness	Delivers 25% more linear-elastic stiffness than conventional glass fiber
Excellent tolerance to damage accumulation	The ability of composite parts to withstand high levels of tension and flexural fatigue without catastrophic failure
S-2 Glass fibers deliver 20% reduction in dielectric constant over E-Glass fibers	Radar transparency
Long shelf life, good machinability and excellent durability	Consistent performance and reliability
Quick wet-out (penetration of resin into the strand)	Faster, more efficient processing
The 933 sizing is stable at processing temperatures of 354°C (670°F)	Facilitates molding with high temperature thermoplastic matrices, yielding exceptional laminate mechanical properties

PRODUCT INFORMATION

Available Products

Yarn Type (metric)	Construction	Nominal Twist TPM	Nominal Twist TPI	Sizing	Approximate Yarn Diameter mm	Approximate Yarn Diameter inch	Nominal Bareglass Yield TEX	Nominal Bareglass Yield Yard/Pound	Denier	Nominal Filament Diameter
SCG75 (SC9 66)	1/0	Z28	0.7Z	933	0.192	0.0076	66	7500	594	"G" or 9 microns

Glass Composition

"S Glass" - reference ASTM C 162-99, ISO 2078, MIL-S-13949H

Solids (% LOI*)

0.10 minimum
0.30 nominal
0.40 maximum
* Loss on ignition after drying

Additional References

Customer acceptance standard: TP-378

Packaging

Package #	7636	
Type Build	Double Taper	
Descriptions	Metric (cm)	English (in)
Inside diameter	6.05	2.38
Tube length	35.6	14.0
Traverse	30.9	12.18
Flange diameter	10.1	3.96
Maximum full package diameter	11.4	4.5
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Quartzel® Yarns

Since 1922, Saint-Gobain Quartz formerly known as Quartz & Silice is a solution provider to Aeronautic, Defense and Railway industries. Based in Nemours (France) and also in Louisville, Kentucky (US), Saint-Gobain Quartz is recognized as world-wide leader in the production of fused quartz fibers which are commercialized under the brand name Quartzel®.

Quartzel® fiber is a homogeneous, non-porous, continuous, amorphous, ultra-pure silica glass with a SiO_2 content ≥ 99.95%. Quartzel® yarns are assemblies of either 9µm, 12.5µm or 14µm filaments gathered into strands (forming package), then processed with varying levels of twist and ply.

Applications

Reinforcement material in composite industry

- Radome (weather, fighter, satcom, DBS, UAVs)
- Electromagnetic windows
- Stealth materials with structural performance for all airborne, grounded & marine military application
- Sacrificial ablative component for thermal protection

Heat insulation at elevated temperature

- Fabrics for furnace lining in industrial and semicon applications
- Thermocouple insulation
- High temperature and fireproof cables
- Thermal protection systems (TPS) of space launchers
- Satellite thermal shield
- Filtration at elevated temperature and/or in aggressive environ.
- High temperature seals
- Insulation in automotive exhausts

Fireproof material for cable industry

- Fireproof and thermal protection for aerospace cables

Main properties

- Outstanding electromagnetic properties thanks to the lowest dielectric constant and dielectric loss among mineral materials (resp. 3.74 and 0.0002 at 10 GHz)
- Non pollutant material with SiO_2 content above 99.95%
- Resistant to most acids
- High mechanical strength & virtually no shrinkage at HT
- Low CTE ($0.54x10^{-6}$ K^{-1}) & good resistance to thermal shock
- Integrity as insulating material up to 1050°C
- Ablative material above 1600°C

Textile processing

Quartzel® yarns are supplied in H3 bobbins and can be used in a large variety of textile processes :

- Weaving / Beaming
- Braiding
- Multiple winding
- Texturizing / Stretch breaking
- Knitting

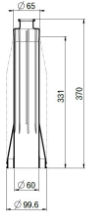
TECHNICAL DATA AND YARN NOMENCLATURE

The following table gives details of standard Quartzel® yarns 9, 12.5 and 14 microns filaments :

Based on ISO 2078 (Europe)	US customary system	Nominal linear density (ISO 1889/ASTM D1907)**		Tensile strength (ISO 3341/ASTM D2256)**	Modulus
		tex	100 x yard/lb	*Typical values in daN****	*Typical values in GPa*
C9 17 Z20 *[sizing*]*[1]	300 1/0 *[sizing*]* 0.5Z[1]	17	300	1.0	
C9 17x2 S150 *[sizing*]*[1]	300 1/2 *[sizing*]* 4Z 3.8S[1]	33	150	2.5	
C9 33 Z20 *[sizing*]*	300 2/0 *[sizing*]* 0.5Z	33	150	2.1	
C14 40 Z20 *[sizing*]*[1]	125 1/0 *[sizing*]* 0.5Z[1]	40	125	2.0	
C12.5 66 Z20 *[sizing*]*	150 2/0 *[sizing*]* 0.5Z	66	75	2.5	
C9 33x2 S150 *[sizing*]*	300 2/2 *[sizing*]* 4Z 3.8S	67	75	5.3	72
C14 80 Z20 *[sizing*]*	125 2/0 *[sizing*]* 0.5Z	80	62	3.8	
C12.5 66x2 S150 *[sizing*]*	150 2/2 *[sizing*]* 4Z 3.8S	132	37	7.5	
C9 33x4 S150 *[sizing*]*[2]	300 2/4 *[sizing*]* 4Z 3.8S[2]	133	37	11.5	
C14 80x2 S150 *[sizing*]*	125 2/2 *[sizing*]* 4Z 3.8S	160	31	10.5	
C9 33x8 S150 *[sizing*]*[2]	300 2/8 *[sizing*]* 4Z 3.8S[2]	266	18	20.0	

[sizing]: Available sizing are : QS1318 (Worldwide) / QSCY1 (Worldwide) / QS13-QPC1 (US Only)*
Standard bobbin weight : 1kg ([1]:500g;[2]:2kg). Carton dimension (mm): L 580 x W 580 x H 400. 15 bobbins max. per carton.
**with a slight internal modification to adapt to quartz material ***Lbf = 2.25 x daN

- QS1318 and QS13-QPC1 are multipurpose high performance direct sizings, recommended for most textile processes and composite applications. They can be used with thermoset resins and among others : Phenolic, Epoxy, BMI, etc..
- QSCY1 sizing is designed specifically for optimized compatibility with cyanate ester resin systems.



H3 bobbins

STORAGE

Quartzel® yarns properties are guaranteed for 24 months when properly stored indoors, protected from direct sunlight exposure and in its original packaging. Store preferably at less than 35°C, avoiding humidity consistently higher than 80%.

https://www.quartz.saint-gobain.com/

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QUARTZ

Quartz
Material datasheet

Quartz is the purest form of glass, it contains above 99,95% of silicon dioxide (SiO_2) This exceptional level of purity which gives outstanding properties to this material.

Among these are:

- *Low thermal expansion with high resistance to thermal shocks*
- *High electromagnetic transparency*
- *High chemical purity and resistance*
- *High softening temperature and thermal resistance*
- *High radiation resistance*

Saint-Gobain Quartz is exclusively using natural Quartz which can be extracted at high purity only from a very limited number of mines on Earth. Our know-how is to process this highly particular material into various forms without altering its original properties. For instance, we are able to process it into: yarn, roving, felt or wool. Further details regarding these particular products can be found in our technical datasheets.

Properties	Description	Value	Units
Physical	Density	2.2	g/cm^3
	Hardness	7	Mohs scale
	Poisson's coefficient	0.16	-
	Ultrasonic wave propagation		
	• Longitudinal :	5960	$m.s^{-1}$
	• Tranversal :	3770	$m.s^{-1}$
	Internal Dampening	0.08	$dBm^{-1}MHz^{-1}$
Electrical	Dielectric constant at 10 GHz	3.74	-
	Loss factor at 10 GHz	0.0002	-
	Dielectric strength	~3.7 x 10^7	$V.m^{-1}$
	Resistivity at 20°C	1 x 10^{20}	Ω.m
	Resistivity at 800°C	6 x 10^8	Ω.m
	Resistivity at 1000°C	1 x 10^8	Ω.m
Thermal	Linear expansion coefficient	0.54 x 10^{-6}	K^{-1}
	Specific heat at 20°C	7.5 x 10^2	$J.kg^{-1}K^{-1}$
	Heat conductivity at 20°C	1.38	$W.m^{-1}K^{-1}$
	Annealing point (log10n = 13)	1220	°C
	Softening point (log10n=7.6)	1700	°C
Optical	Refractive index	1.4585	-
	Dispersion	67	-
	Field of transparency	0.2 to 4	µm