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SOUR FLUID IMMERSION TESTING OF THERMOPLASTIC MATERIALS ACCORDING TO API 6A (ISO 10423 APPENDIX F.1.13.5.2)

Version Control

Revision	Date	Description of Change + Reason for Change	Prepared by	Reviewed by
Initial issue	17-NOV-2022	FIRST ISSUE	K.SOMANI	M.LEWAN

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1. SYNOPSIS

Sour fluid immersion testing of four non-metallic materials – KIPTFE01, KITFM03, KIVPT01 and KICFT02 from Industrial Spares Manufacturing & Trading Co. (ISMAT) has been undertaken according to API6A/ISO 10423 F.1.13.5.2. The exposure temperature was 177 °C and soak duration 160 hours with 1000 psi (69 bar) of a gas mixture containing 10% H_2S (class FF/HH). Performance was evaluated by measuring changes in mass, volume, hardness and tensile property levels, all at room temperature.

The grid below summarises material performance.

Material	Mean change (%) after Immersion				
	Mass	Volume	Young's Modulus	Maximum Stress	Elongation at Break
KIPTFE01	1.32	2.23	-27	-13	-6
KITFM03	1.30	3.17	-42	2	8
KIVPT01	1.12	2.95	-26	-14	2
KICFT02	1.61	2.40	-36	-15	15

All specimens of all four materials were intact after the sour fluid exposure, with no visible evidence of chemical ageing, and none was expected. No acceptance criteria are defined in API 6A. By this measure, all four materials performance in this short-term high temperature sour fluid exposure test is considered suitable at test conditions.

2. OBJECTIVES

Industrial Spares Manufacturing & Trading Co. (ISMAT) have contracted Element Hitchin to carry out testing of four thermoplastics according to API 6A (ISO10423:2009[1]), Appendix F1.13.5.2. The test fluid is multi-phase with the testpieces located in the hydrocarbon oil phase. ISMAT specified class FFHH.

3. MATERIALS AND METHOD

Ismat delivered KIPTFE01, KITFM03, KIVPT01 and KICFT02 to Element Hitchin on 9th August 2022 in the form of strips of dimension 1000 mm × 3.5 mm in thickness and 50 mm in width. As received materials were logged with a unique reference number for quality and traceability purposes (Table 3.1).

[1] Petroleum and natural gas industries – Drilling and production equipment – Wellhead and Christmas tree equipment" section F.1.13.5.2, class FF/HH.

Table 3.1: Test material received for testing

Test material	Description	Element Quality Reference Number	QUANTITY AND FORM	BATCH AND LOT NUMBER
KIPTFE01	15% GLASS FILLED PTFE NFF	M28159	1off 1000 mm×50mm× 3mm strip	Not disclosed
KITFM03	MODIFIED PTFE	M28160	1off 1000 mm×50mm× 3mm strip	Not disclosed
KIVPT01	VIRGIN PTFE FF	M28161	1off 1000 mm×50mm× 3mm strip	Not disclosed
KICFT02	25% CARBON FILLED PTFE NFF	M28162	1off 1000 mm×50mm× 3mm strip	Not disclosed

The bag labels and representative specimens were photographed and are shown in Appendix A. From the supplied strip Element Hitchin machined 10 off ASTM 638 size type IV dumbbells each. Five of the ten tensile dumbbells were used for control testing and the remaining five for sour fluid exposure. Three 50 mm×25mm×3mm rectangular specimens were used for mass/volume/hardness (MVH) measurements. The sour gas mixture was procured from Compressed Gas solutions Ltd, the certificate of composition is shown in Appendix B. All chemicals were procured from Fisher Scientific UK.

3.1 Test conditions

The exposure test conditions are summarised in Table 3.2

Table 3.2 : ISO 10423 – F.1.13.5.2 – test conditions

Temperature	180± 2 °C
Pressure	1000 psi (69 bar)
Gas phase	FF/HH: 10/80/10 mol% $H_2S/CO_2/CH_4$
Liquid phases	5% volume of water (deionised water, conductivity < 5μS) + 60% volume of NORSOK oil (mixtures of 70% heptane, 20% cyclohexane, 10% toluene)
Replication	5
Exposure time	160 hours, minimum

3.2 Test procedure

The test samples of all four thermoplastic materials were exposed together in the hydrocarbon oil phase of the fluid. The immersion test was carried out in a pressure vessel (Figure 3.1), equipped with an external band heater, an internal thermocouple, a calibrated pressure sensor and an isolation needle valve. Pressure and temperature were logged throughout by a PC running dedicated data acquisition software.

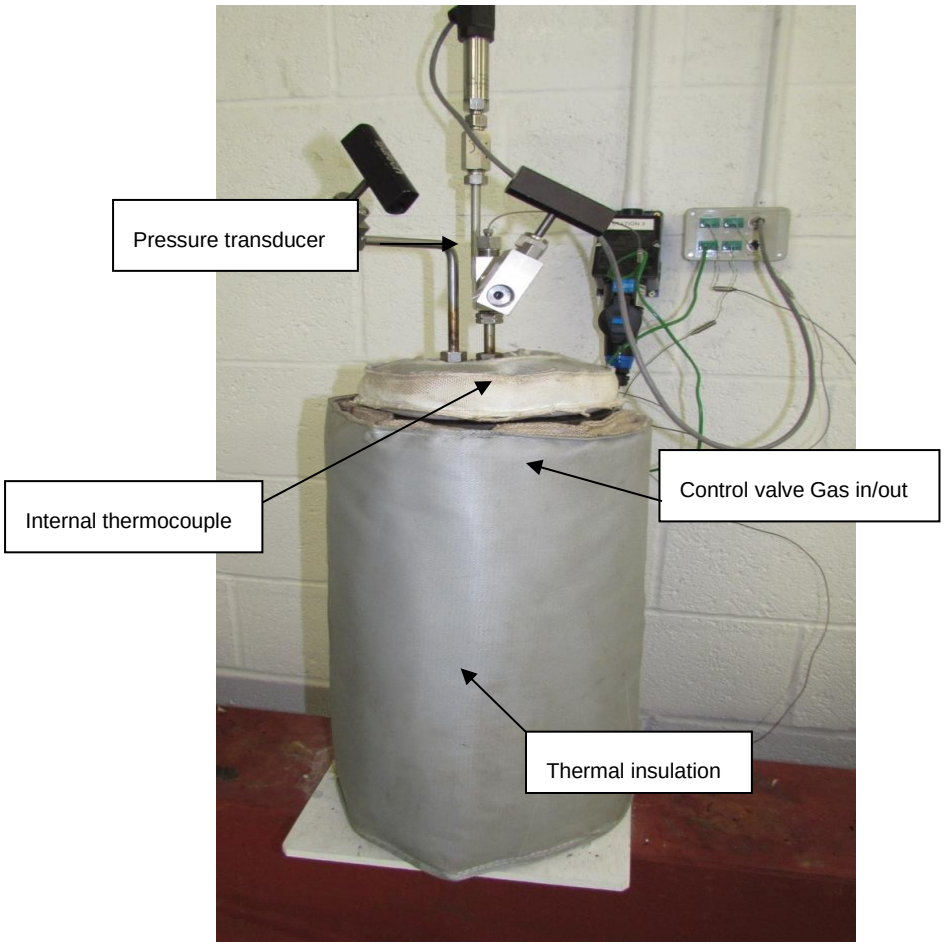

Figure 3.1: Pressure vessel for sour fluid test equipped with pressure sensor, internal thermocouple and needle valve.

After placing the test samples and required volume of test liquids into the pressure vessel, the cell was closed and flushed with nitrogen to remove air (oxygen), then heated to 180°C. The test gas mixture 10/80/10 vol% $H_2S/CO_2/CH_4$ was added from the supplied cylinder to 1000 psi ± 100 psi (75 bar) using a dedicated booster pump. During the test, the gas pressure slowly reduced due to absorption of gas into the liquids and samples. This resulted in the gas pressure having to be boosted once during the test. After 160 hours at test temperature and pressure, the vessel was cooled naturally to ambient temperature, which led to a pressure decrease. The samples remained in the vessel under these conditions overnight before the gas was released at a slow rate of 1 bar/minute (14 psi/minute). Samples were left in the pressure vessel undisturbed overnight before being retrieved after flushing with nitrogen. They were weighed and measured for hardness 30 minutes later.

Tensile testing was carried out in accordance with ASTM D638. Young's modulus was calculated from the stress strain curves using Zwick software. The testing employed a Zwick Z050 screw-driven test machine equipped with a calibrated 50 kN load cell. A contacting arm extensometer was used for strain measurement. Test speed was 50 mm/minute.

A calibrated milligramme electronic balance was used for all weighings. The tensile samples were stored in fresh NORSOK oil until tensile testing could be performed. A calibrated Shore D hardness durometer was used to measure hardness.

4. RESULTS

Appendix C, Figure C.1 shows the pressure/temperature plot for the immersion test at 180°C. The test was run between 4th October to 12th October 2022. Photographs of all the exposed samples are shown in Appendix D.

Table 4.1 lists the changes in mass, volume and hardness measured for the thermoplastics after 160 hours at 180°C. The quoted values are the mean of three measurements.

Table 4.1: Mass, volume and hardness change of thermoplastics after immersion testing

Compound	Element Material Reference Number	Replication	After immersion			Mean change		
			Mass Change (%)	Volume change (%)	Hardness Shore D Units	Mass Change (%)	Volume change (%)	Hardness Shore D Units
KIPTFE01	M28159	1	1.32	2.07	-6	1.32	2.23	-6
		2	1.29	2.23	-6			
		3	1.34	2.39	-6			
KITFM03	M28160	1	1.32	3.21	-11	1.30	3.17	-11
		2	1.25	3.02	-10			
		3	1.33	3.27	-11			
KIVPT01	M28161	1	0.95	2.64	-10	1.12	2.95	-10
		2	1.18	2.97	-9			
		3	1.24	3.23	-10			
KICFT02	M28162	1	1.57	2.31	-9	1.61	2.40	-9
		2	1.60	2.38	-10			
		3	1.66	2.52	-9			

Initial Shore D hardness of KIPTFE 01, KITFM03, KIVPT01 and KICFT02 were 59, 57, 54 and 54 Shore D units respectively. Change in mass and volume of all five materials is small. Mass and volume changes (%) of all materials are shown graphically in Figure 4.1. Hardness of KITFM03 and KIVPT01 deceased more than 10 units after immersion. Absorbed fluid is responsible for change in physical properties.

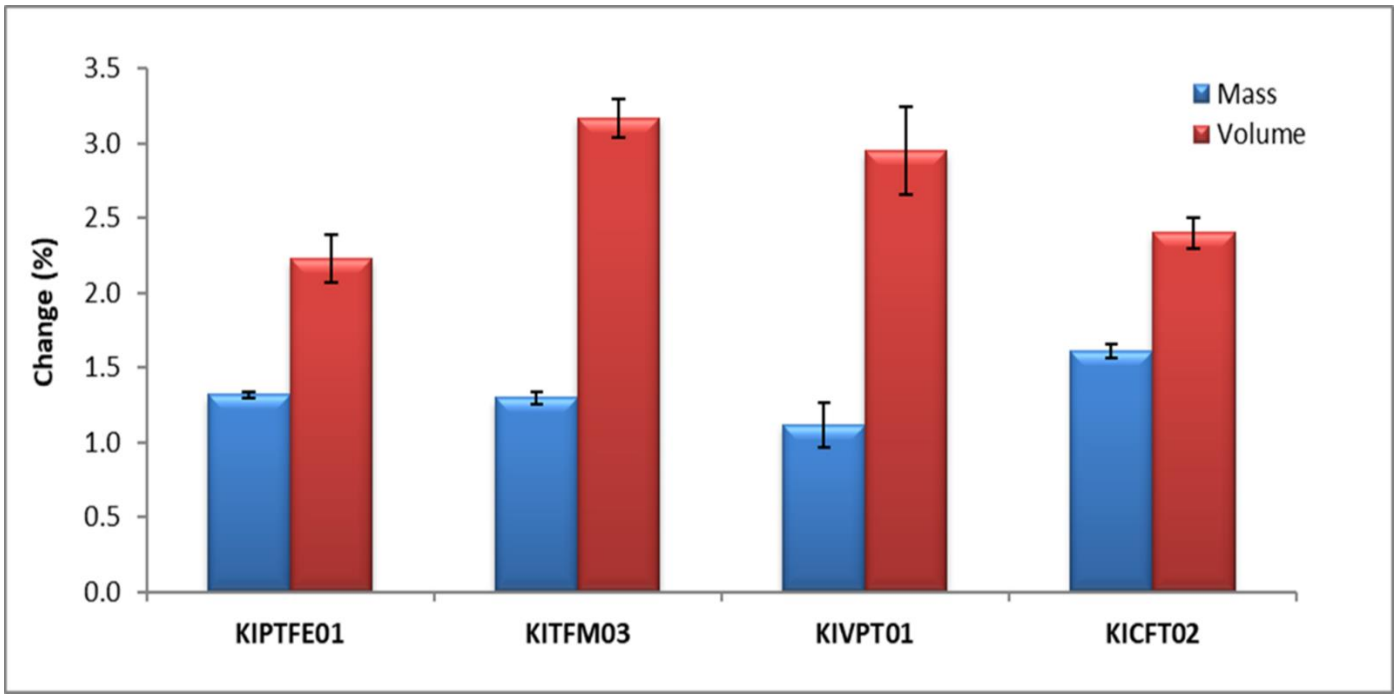

Figure 4.1: **Mass and volume changes (%) of all four material after 160 hours immersion at 180 °C/69 bar.**

The tensile data before and after testing is summarizsed in Table 4.2; the quoted values are the mean of 5 results. Tensile properties of materials are plotted and shown in Figure 4.2. Stress vs strain curves of all specimens before and after immersion testing are in Appendix E.

The presence of absorbed liquid is responsible for the measured changes in tensile properties of all four materials. There was no visual change apparent or any evidence of degradation after the immersion test.

Table 4.2: Young's modulus, tensile strength and elongation at break data for KIPTFE01, KITFM03, KIVPT01 and KICFT02 before and after immersion

Material ID	Condition	Young's modulus	% Change	Yield Stress	% Change	Yield strain	% Change	Maximum Stress	% Change	Elongation at Break	% Change
		GPa		MPa		%		MPa		%	
KIPTFE01	Unaged	0.25	-27	-	-	-	-	20.52	-13	263	-6
	Fluid Exposed	0.18		-		-		17.78		247	
KITFM03	Unaged	0.27	-42	11.84	-16	56	35	22.57	2	383	8
	Fluid Exposed	0.16		9.92		75		23.00		415	
KIVPT01	Unaged	0.26	-26	-	-	-	-	25.60	-14	341	2
	Fluid Exposed	0.19		-		-		22.07		346	
KICFT02	Unaged	0.26	-36	-	-	-	-	14.56	-15	192	15
	Fluid Exposed	0.17		-		-		12.43		220	

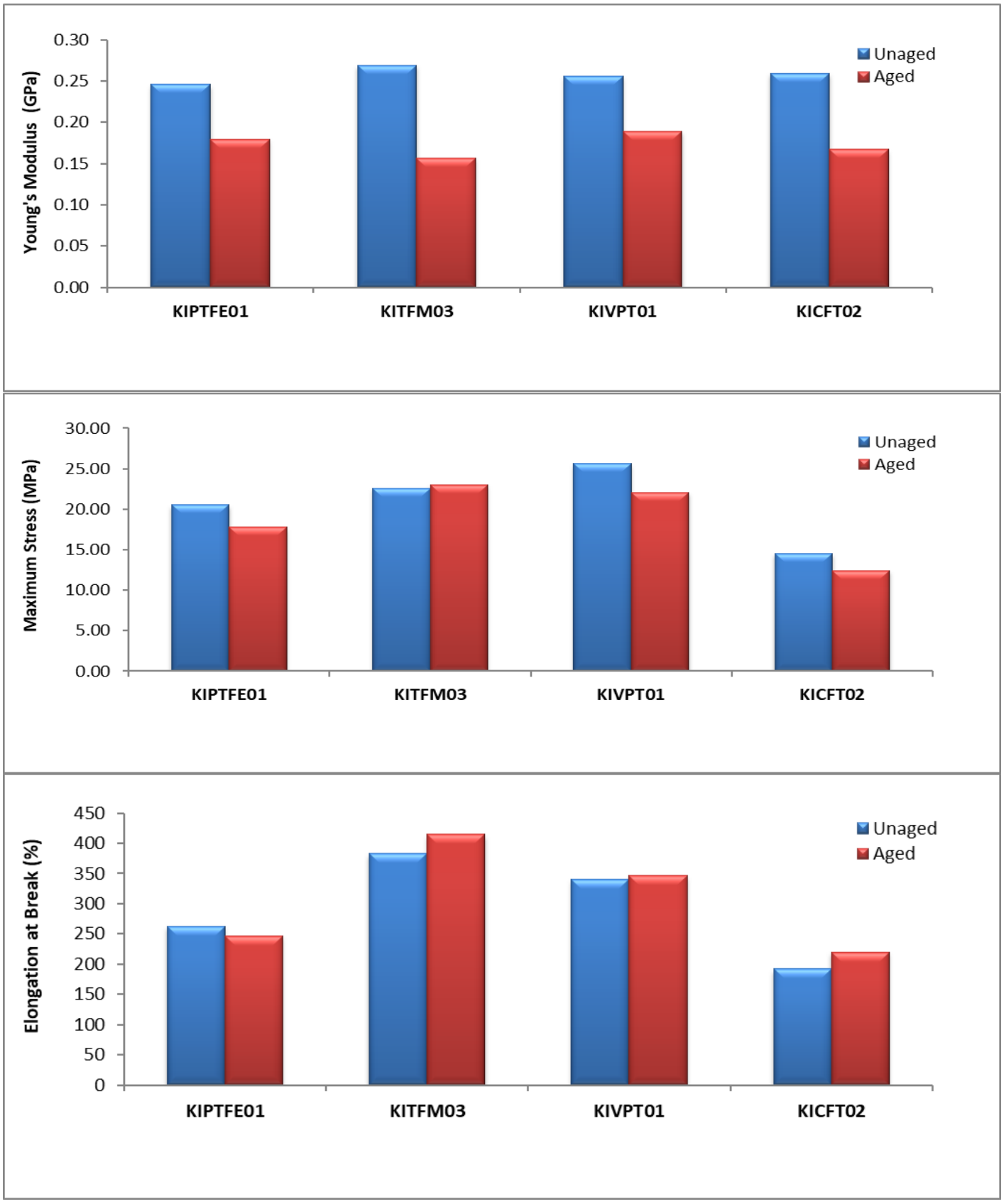

Figure 4.2: Tensile properties of all thermoplastic materials before and after immersion

5. CONCLUSIONS

The behaviour of all thermoplastics materials- PTFE's from ISMAT was as expected after exposure in a multi-phase sour fluid at 180°C for a week. Chemical ageing of this polymer was not expected in these conditions and no evidence of deterioration was found. The grid below summarises material performance.

Material	Mean change (%) after Immersion				
	Mass	Volume	Young's Modulus	Maximum Stress	Elongation at Break
KIPTFE 01	1.32	2.23	-27	-13	-6
KITFM03	1.30	3.17	-42	2	8
KIVPT01	1.12	2.95	-26	-14	2
KICFT02	1.61	2.40	-36	-15	15

APPENDIX A As- received samples

Figure A.1: As-received KIPTFE01

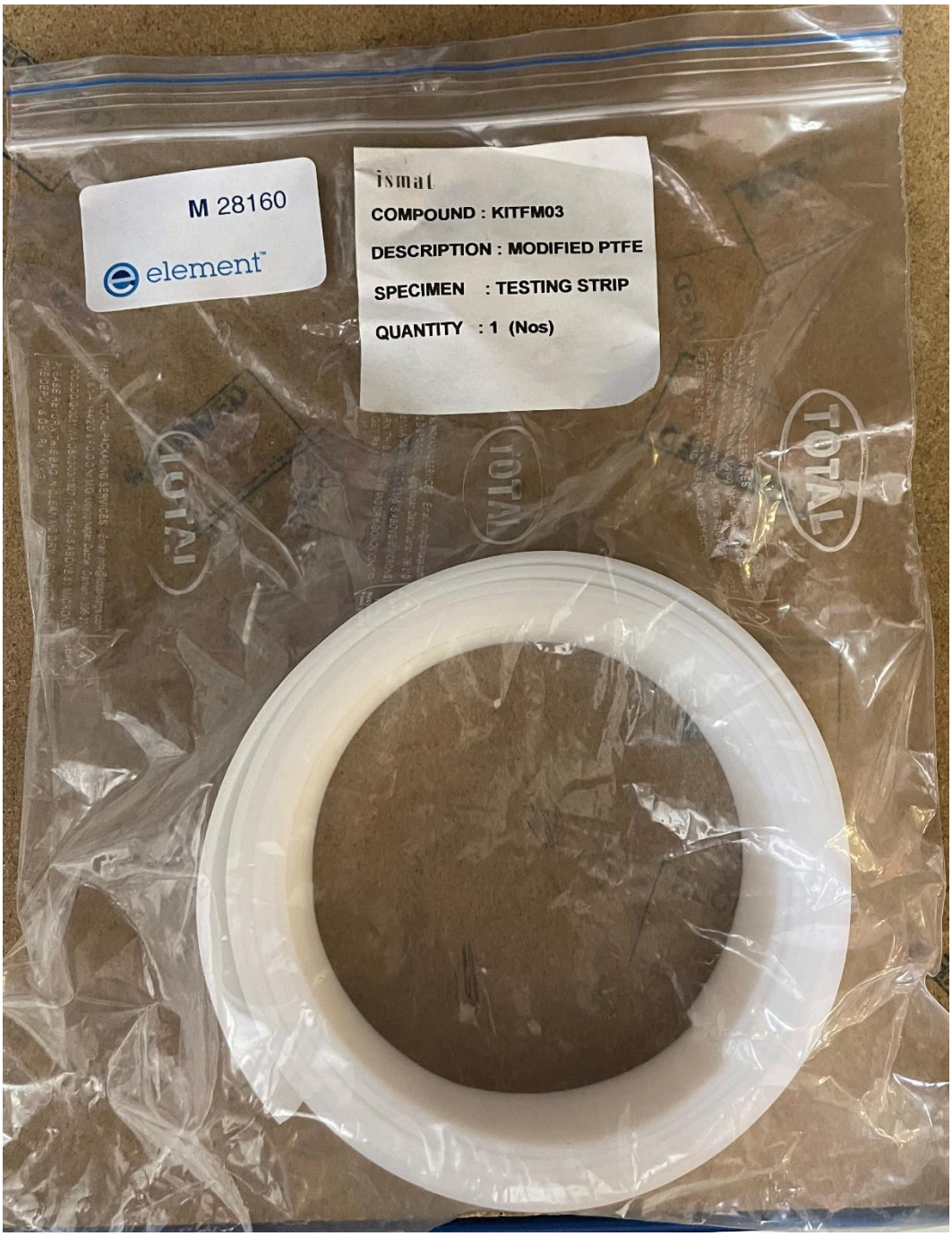

Figure A.2: As-received KITFM03

Figure A.3: As-received KIVPT01

Figure A.4: As-received KICFT02

APPENDIX B Gas certificate

ELEMENT HITCHIN

Compressed Gas Solutions Ltd

CYLINDER TYPE	50L STEEL	**DATE**	06/09/2022
VALVE TYPE	BS15	**CGS REF**	2022-2510
STABILITY	06/09/2024	**CUSTOMER REF**	GB10100611PO-2
CERTIFIED BY	PL		
PRESSURE	32 BAR		
VOLUME	2.0 M3		
NET WEIGHT	3.3 KG		

CYLINDER NO.

13710774

CERTIFICATE OF COMPOSITION

COMPONENT	REQUESTED VALUE	CERTIFIED VALUE
HYDROGEN SULPHIDE 2.5	10.0%	10.0%
METHANE 3.5	10.0%	9.97%
CARBON DIOXIDE 4.5	BALANCE	BALANCE

ALL UNITS ARE MOLAR, WITH A MIXTURE ACCURACY OF ±2%

KEEP THE MIXTURE ABOVE 0°C TO PREVENT CONDENSATION OF THE CONDENSABLE PRODUCTS, IF PRESENT.

PRODUCTS ARE FILLED GRAVIMETRICALLY AND TRACEABLE TO STANDARDS 218M CALIBRATED AT THE NATIONAL PHYSICAL LABORATORY, TEDDINGTON.

Compressed Gas Solutions Ltd, J Reid Trading Estate, Factory Road, Sandycroft, Flintshire, CH5 2QJ, +44 (0) 1244 520688

Figure B.1: Test gas mixture certificate: 10/80/10 mol% H2S/CO2/CH4

APPENDIX C Temperature and pressure vs time plot

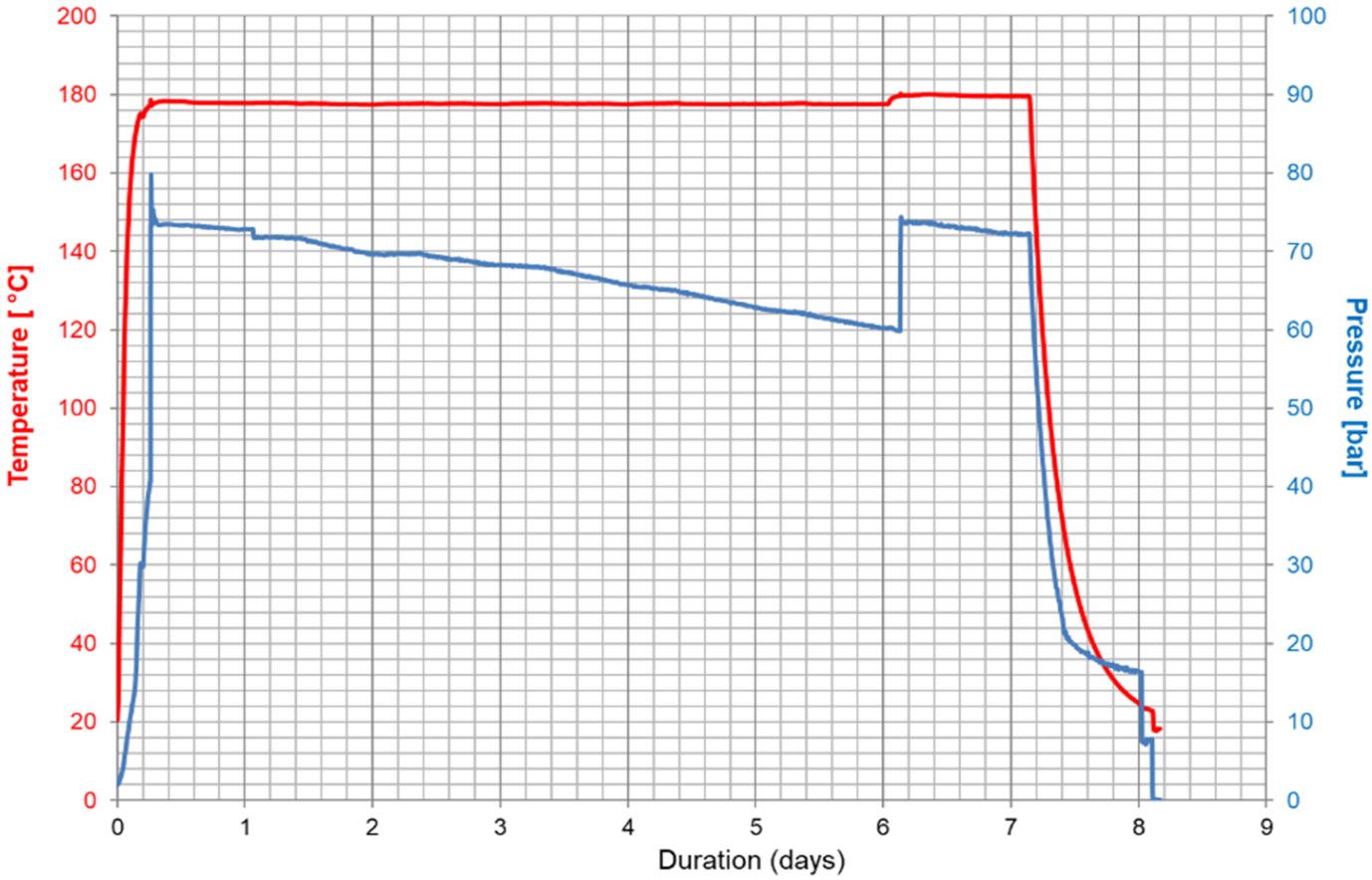

Figure C.1: Pressure-Temperature plots for test exposure

APPENDIX D Samples before and after immersion

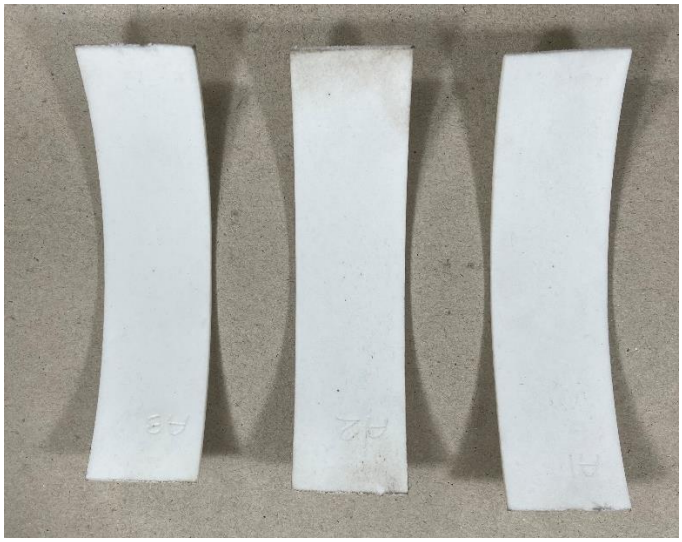

Figure D.1: KIPTFE01 before immersion

Figure D.2: KITFM03 before immersion

Figure D.3: KIVPT01 before immersion

Figure D.4: KICFT02 before immersion

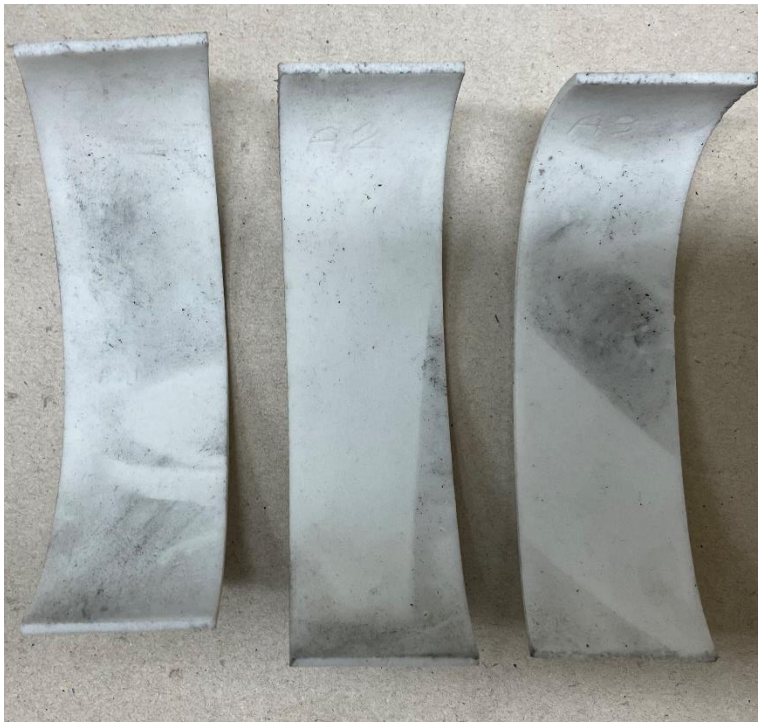

Figure D.5: KIPTFE01 after immersion

Figure D.6: KITFM03 after immersion

Figure D.7: KIVPT01 after immersion

Figure D.8: KICFT02 after immersion

APPENDIX E Stress vs strain curve of all materials

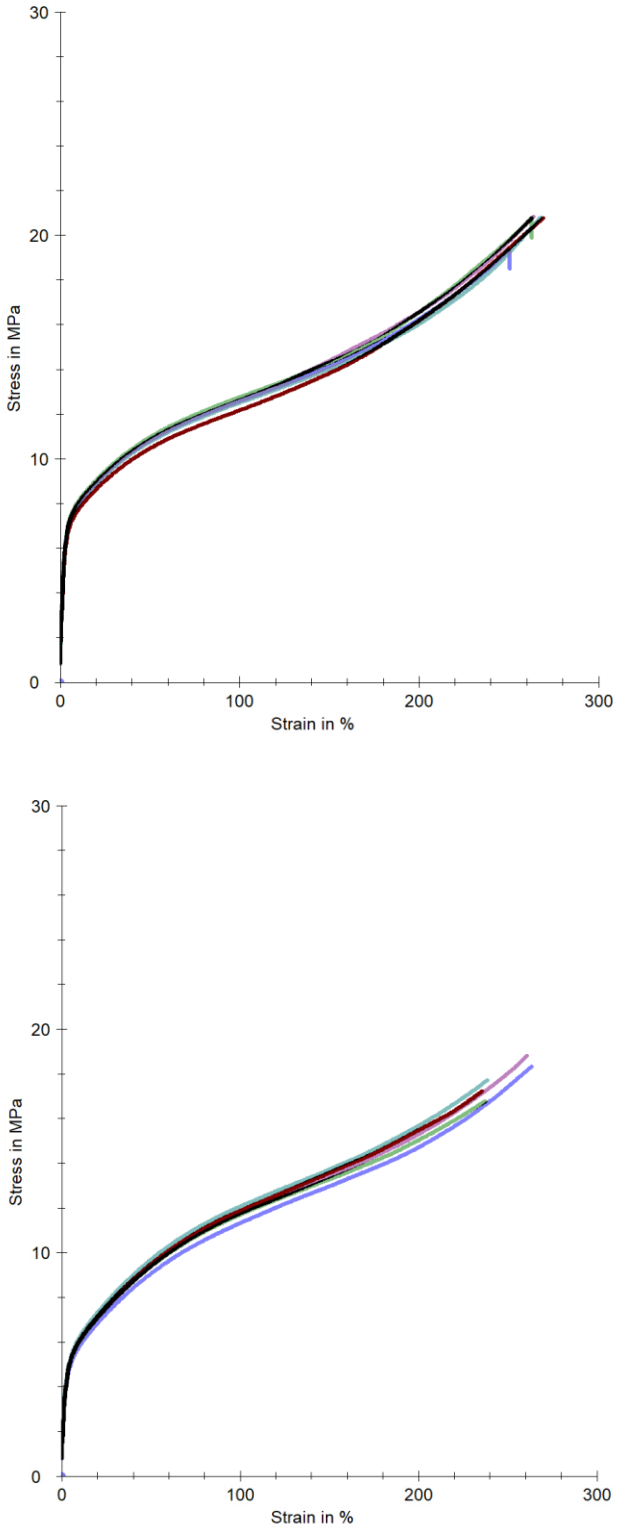

Figure E.1 Stress vs. strain curves of KIPTFE01 before (above) and after (below) immersion

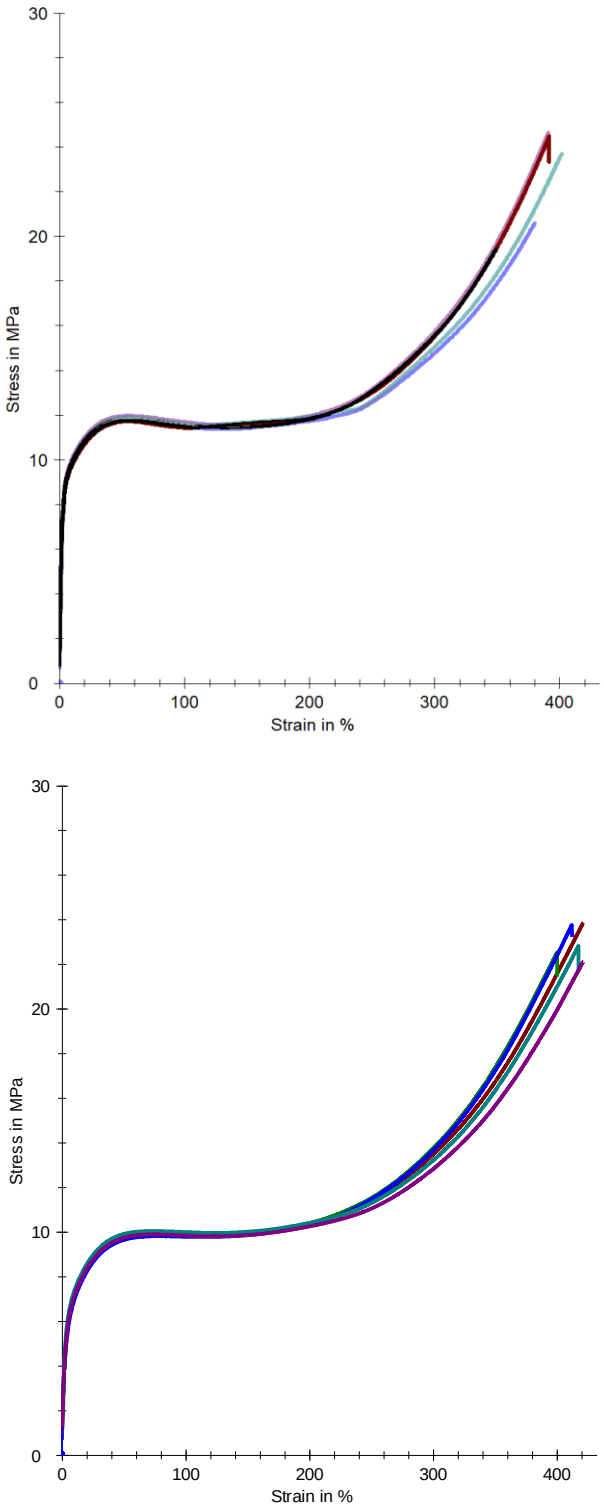

Figure E.2 Stress vs. strain curves of KITFM03 before (above) and after (below) immersion

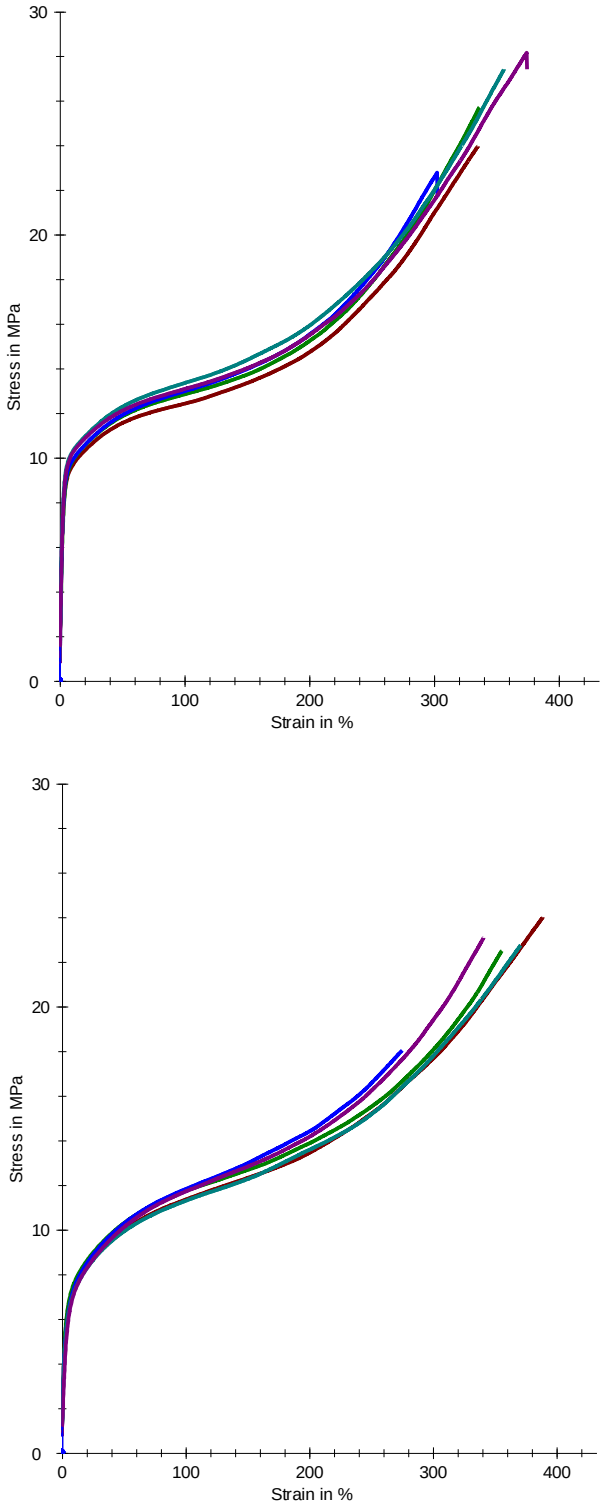

Figure E.3 Stress vs. strain curves of KIVPT01 before (above) and after (below) immersion

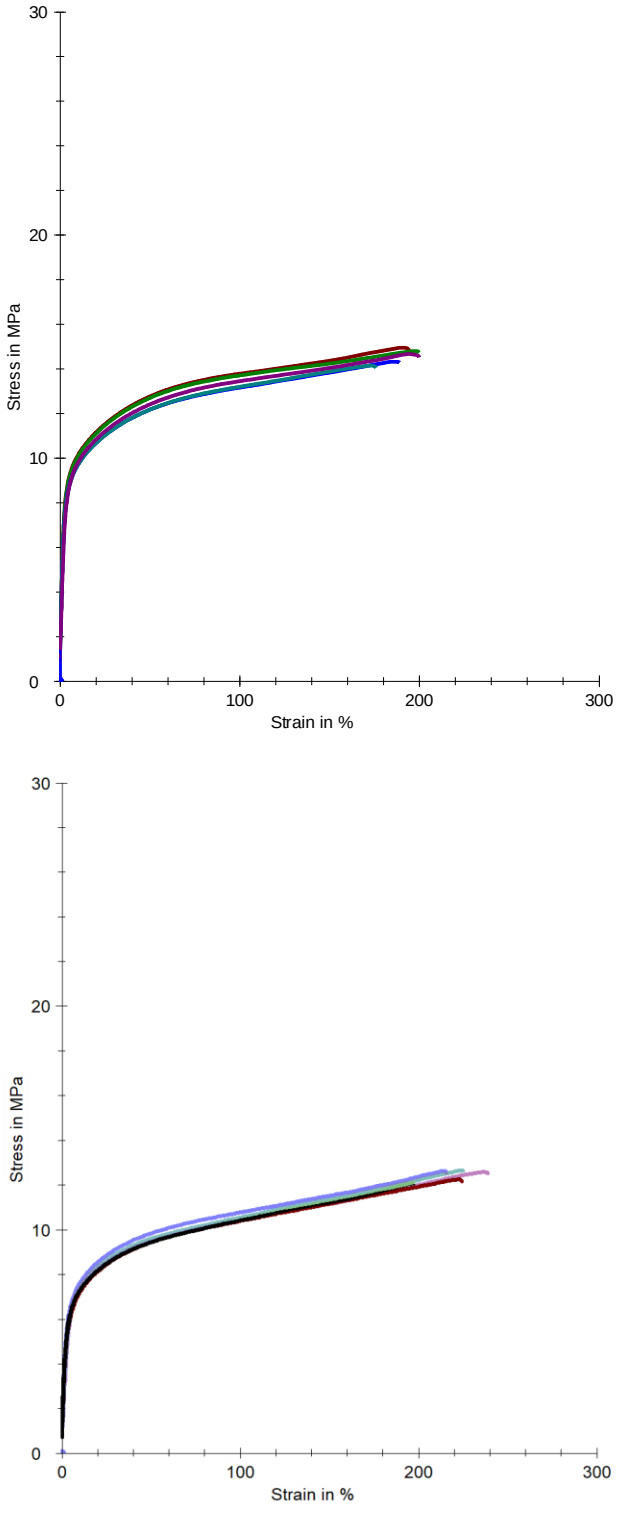

Figure E.4 Stress vs. strain curves of KICFT02 before (above) and after (below) immersion