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SOUR FLUID IMMERSION TESTING OF ELASTOMERIC 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 three fluoroelastomeric materials – KIFK10, KIFK11 and KIFK15 from Industrial Spares Manufacturing & Trading Co. (ISMAT) has been undertaken according to API6A/ISO 10423 F.1.13.5.2. The exposure temperature was 200 °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.

Property	Measured change after immersion		
	KIFK10	KIFK11	KIFK15
Volume (%)	10.6	11.6	10.7
Hardness (Shore A, unit)	-11	-7	-11
Modulus at 50% elongation (%)	-49	-41	-36
Tensile Strength (%)	-21	-56	-41
Elongation at Break (%)	+29	-14	-9

2. OBJECTIVES

Industrial Spares Manufacturing & Trading Co. (ISMAT) have contracted Element Hitchin to carry out testing of three elastomeric materials 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.

This document summarises the work undertaken by Element Hitchin.

3. MATERIALS AND METHOD

Ismat delivered KIFK10, KIFK11 and KIFK15 to Element Hitchin on 9th August 2022 in the form sheet 150 mm×150 mm × 2 mm. 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
KIFK10	FKM GLT AED -30C	M28163	2 off 150 mm×150mm× 2mm SHEET	Not disclosed
KIFK11	FKM AED -18C	M28164	2 off 150 mm×150mm× 2mm SHEET	Not disclosed
KIFK15	FKM GLT AE1 AED -46C	M28165	2 off 150 mm×150mm× 2mm SHEET	Not disclosed

The bag labels and representative specimens were photographed and are shown in Appendix A. From supplied sheet 10 tensile dumbbells of ISO 37 type 2 were stamped from the sheets of each material by Element using a certified cutter; the level of replication was five for the fluid exposure. Also, three replicate rectangles of size 5 cm × 2.5 cm were cut for mass/volume/hardness (MVH) measurements.

Five of the ten tensile dumbbells were used for control testing and the remaining five for sour fluid exposure. 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	200± 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 three elastomeric material 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.

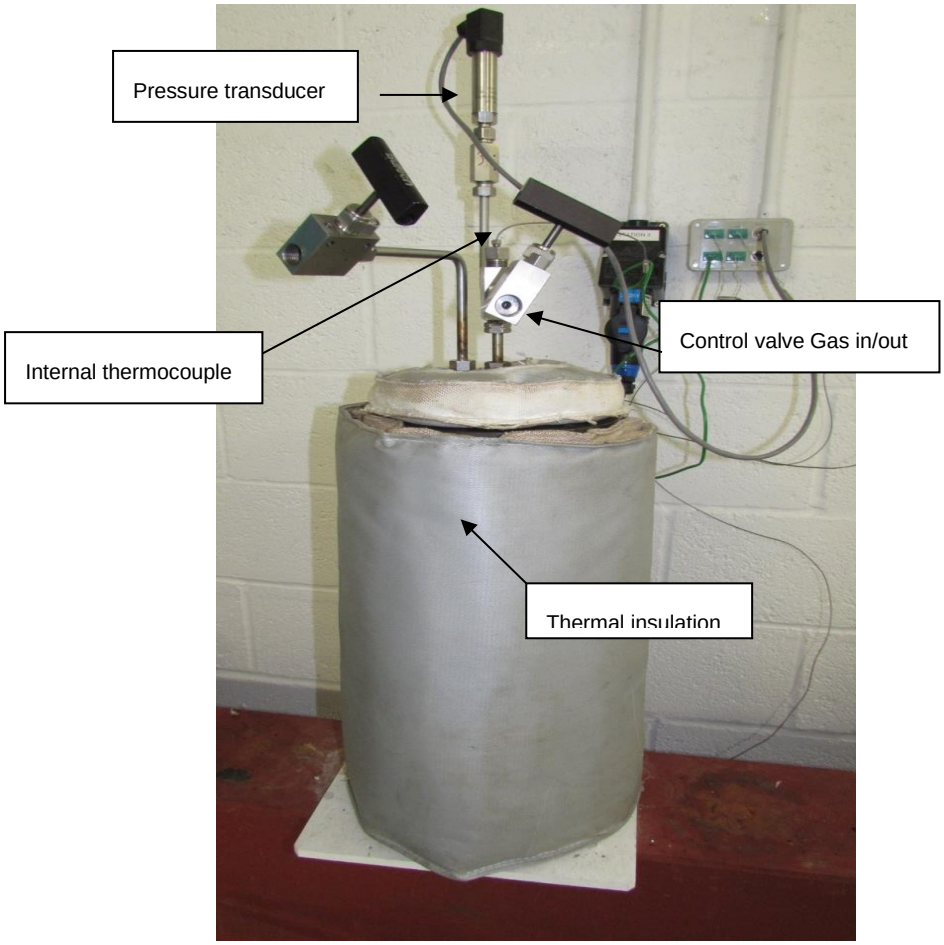


Figure 3.1: Pressure vessel for sour fluid test equipped with pressure sensor, internal thermocouple and needle valve.

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 200 °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 ISO 37. The testing employed a Zwick Z050 screw-driven test machine equipped with a calibrated 50kN load cell. A contacting arm extensometer was used for strain measurement. Test speed was 500 mm/minute.

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

4. RESULTS

Appendix C, Figure C.1 shows the pressure/temperature plot for the immersion test at 200°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 elastomeric materials after 160 hours at 180°C. The quoted values are the mean of three measurements.

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

Compound	Element Material Reference Number	Replication	After immersion			Mean change		
			Mass Change (%)	Volume change (%)	Hardness Shore A (Units)	Mass Change (%)	Volume change (%)	Hardness Shore A (Units)
KIFK10	M28163	1	4.07	10.35	-11	4.25	10.6	-11
		2	4.22	10.50	-11			
		3	4.45	10.86	-11			
KIFK11	M28164	1	3.36	11.45	-7	3.34	11.6	-7
		2	3.48	12.02	-7			
		3	3.18	11.33	-7			
KIFK15	M28165	1	4.06	10.54	-11	4.05	10. 7	-11
		2	4.14	10.70	-11			
		3	3.94	10.76	-12			

Initial hardness of KIFK10, KIFt11 and KIFK15 were 87, 88 and 86 Shore A units respectively.

Mass and volume change (%) of all elastomers is shown graphically in Figure 4.1. All 3 elastomers had similar mass and volume changes. Element Hitchin is not aware of the polymer type of any of the tested materials. All three-materials hardness reduced after immersion.

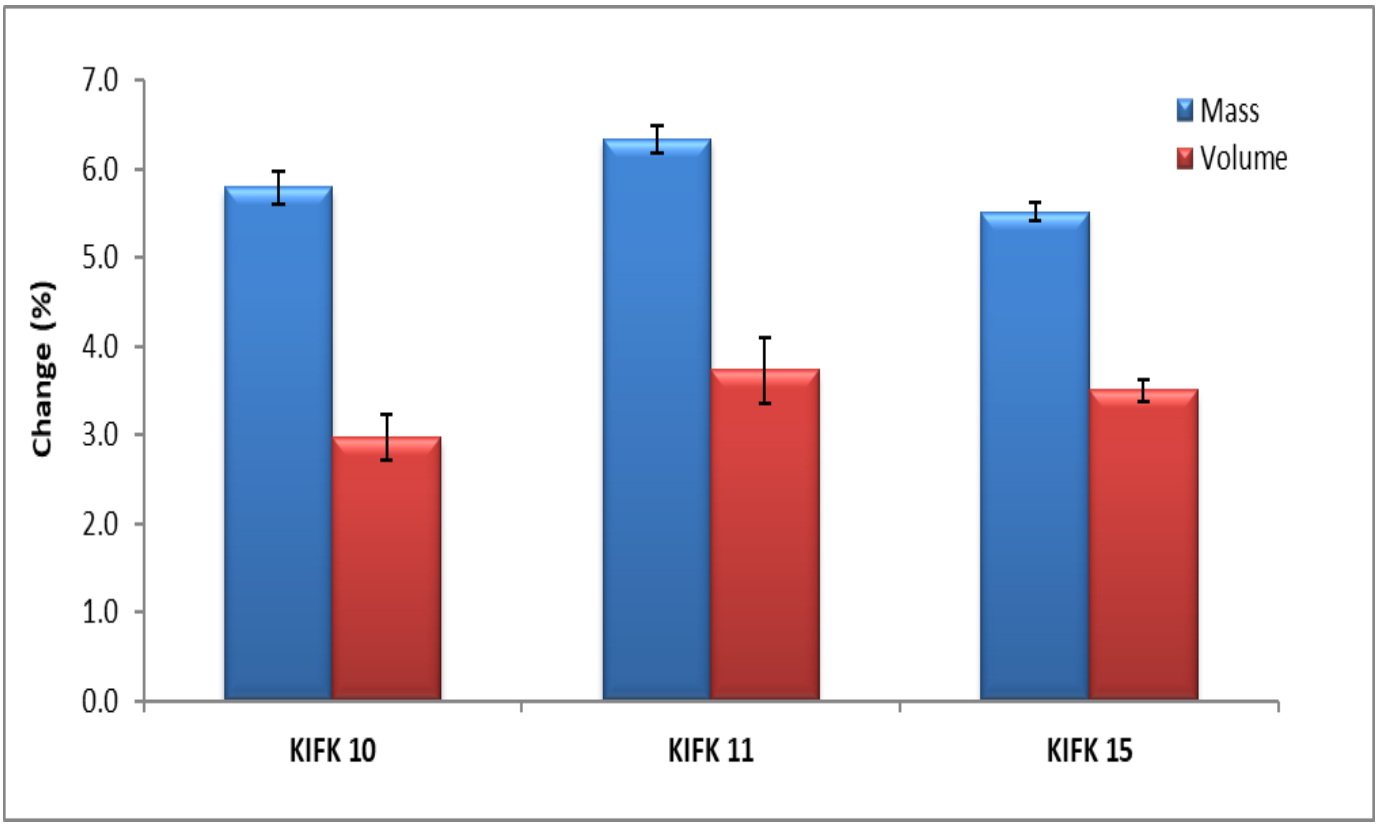


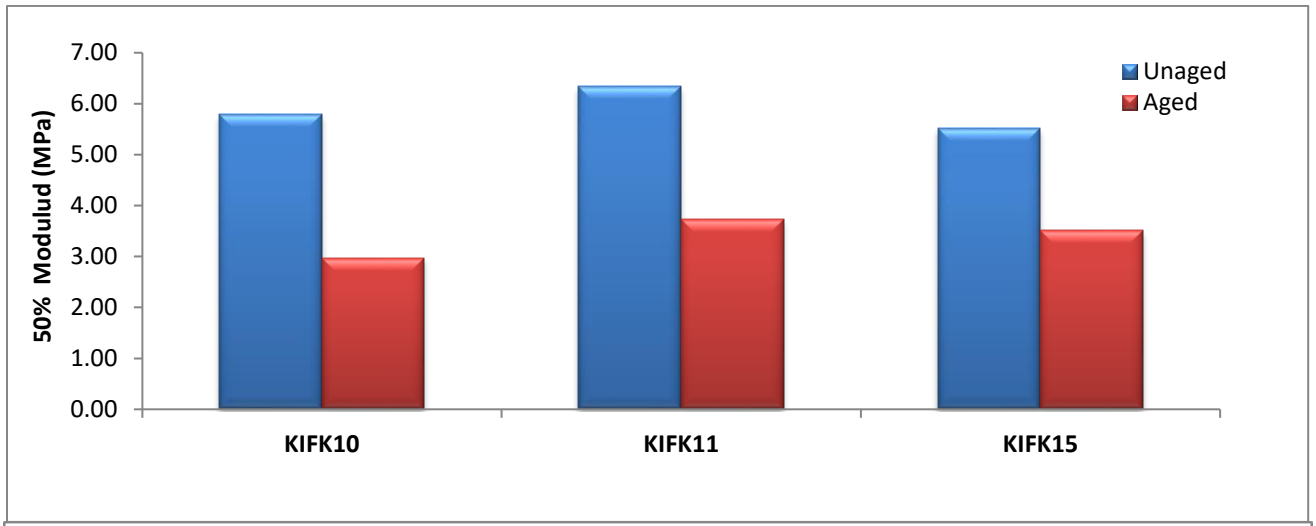
Figure 4.1: **Mass and volume changes (%) of all three materials after 160 hours immersion at 200 °C/69 bar.**

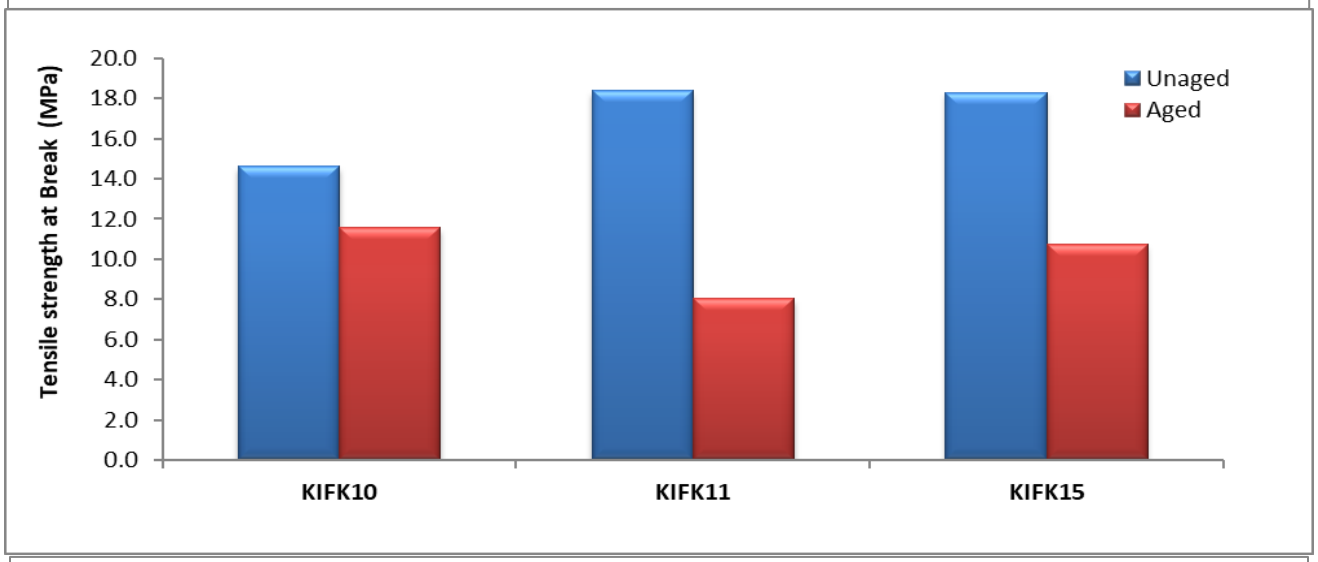
The tensile data before and after testing is summarised in Table 4.2; the quoted values are the mean of 5 results. Tensile properties of materials are plotted and shown in Figure 4.2. Stress vs strain curves of all specimens before and after immersion testing are shown in Appendix E.

Tensile dumbbells and MVH specimens of all elastomers were in visibly good condition after the immersion, no signs of blisters or cracks.

Table 4.2: Tensile properties of elastomeric materials before and after immersion

Material ID	Condition	Modulus at 50% strain	% Change	Modulus at 100% strain	% Change	Tensile strength at Break	% Change	Elongation at Break	% Change
		MPa		MPa		MPa		%	
KIFK10	Unaged	5.79	-49	13.87	-40	14.61	-21	107	29
	Aged	2.97		8.30		11.58		138	
KIFK11	Unaged	6.34	-41	11.81	-46	18.38	-56	163	-14
	Aged	3.73		6.39		8.03		139	
KIFK15	Unaged	5.52	-36	12.77	-30	18.29	-41	135	-9
	Aged	3.50		8.89		10.73		123	





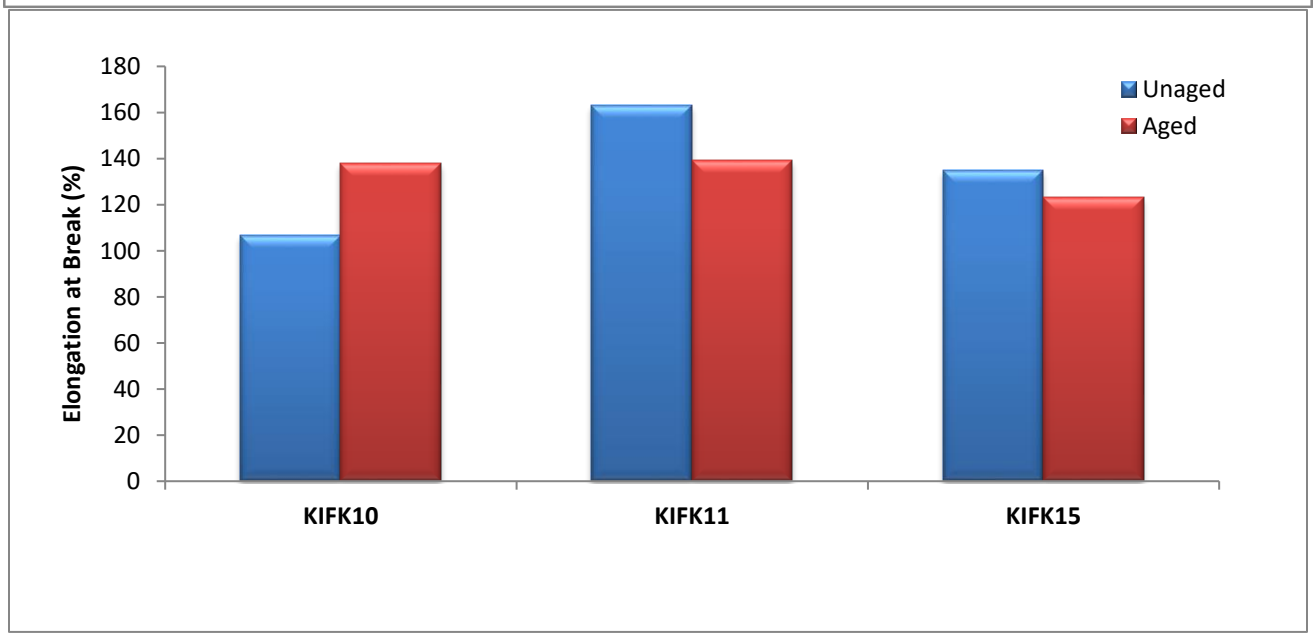


Figure 4.2: Tensile properties of all three elastomeric materials before and after immersion

There was a moderate to large reduction in modulus, tensile strength and elongation at break for both KIFK11 and KIFK15. KIFK10 had similar levels of reduction in modulus and tensile strength, but elongation at break increased.

5. CONCLUSIONS

The behaviour of all three fluoro elastomeric materials from ISMAT was as expected after exposure in a multi-phase sour fluid at 200 °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.

Property	Measured change after immersion		
	KIFK10	KIFK11	KIFK15
Volume (%)	10.6	11.6	10.7
Hardness (Shore A, unit)	-11	-7	-11
Modulus at 50% elongation (%)	-49	-41	-36
Tensile Strength (%)	-21	-56	-41
Elongation at Break (%)	+29	-14	-9

APPENDIX A As- received samples

Figure A.1: As-received KIFK10

Figure A.2: As-received KIFK11

Figure A.3: As-received KIFK15

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% $H_2S/CO_2/CH_4$

APPENDIX C Temperature and pressure vs time plot

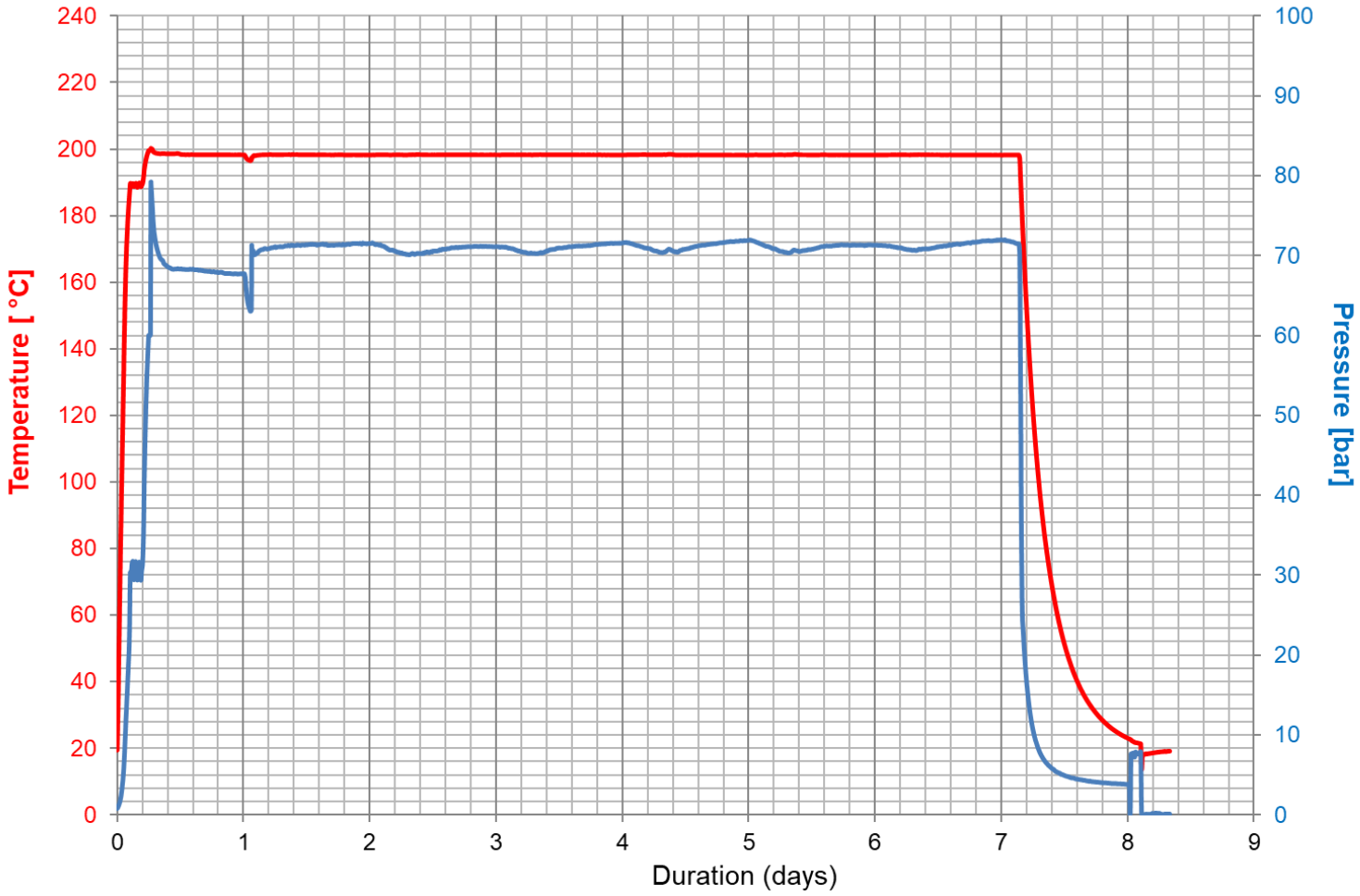


Figure C.1: Pressure-Temperature plots for test exposure

APPENDIX D Samples before and after immersion

Figure D.1: KIFK10 before immersion

Figure D.2: KIFK11 before immersion

Figure D.3: KIFK15 before immersion

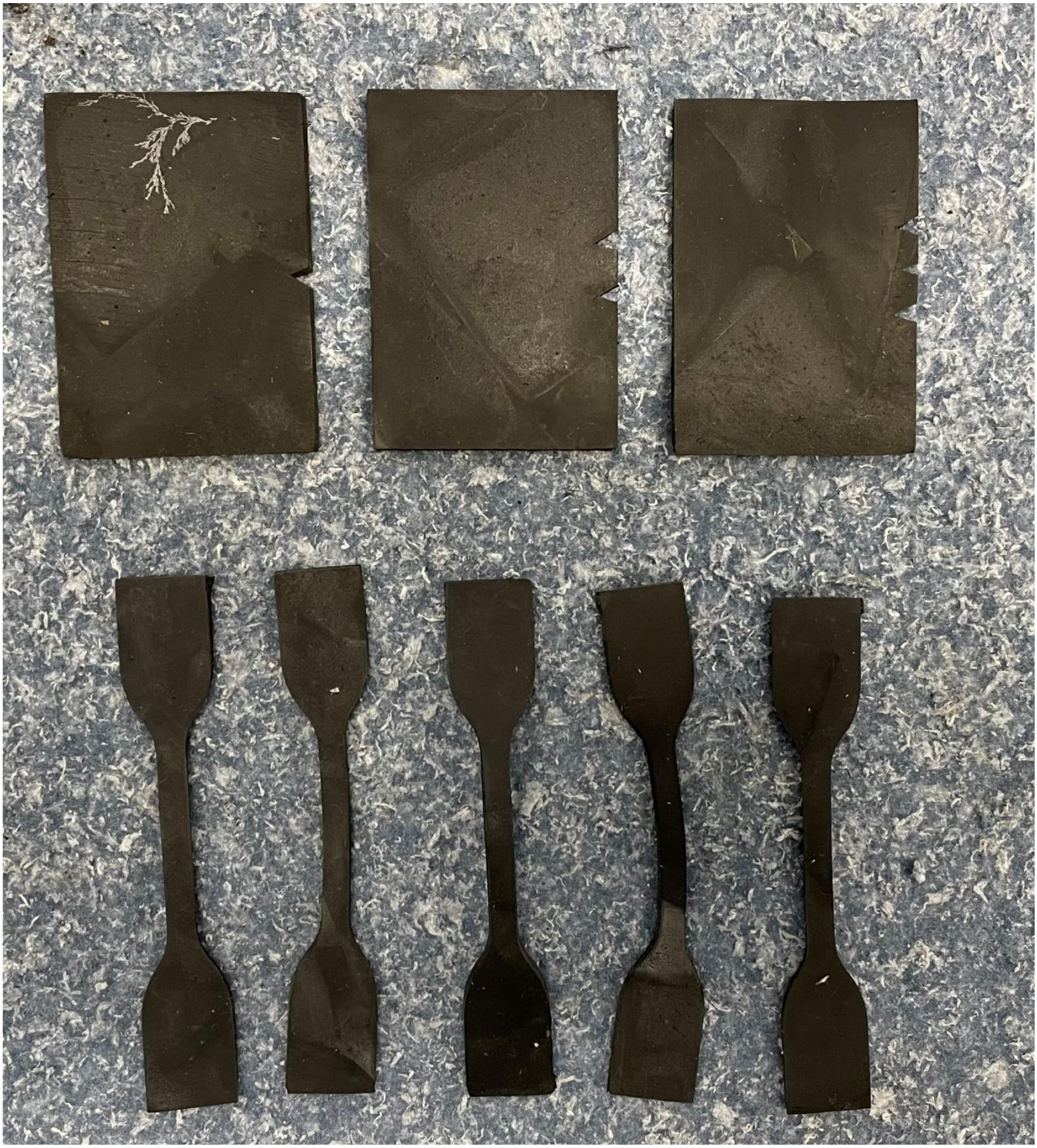


Figure D.4: KIFK10 after immersion

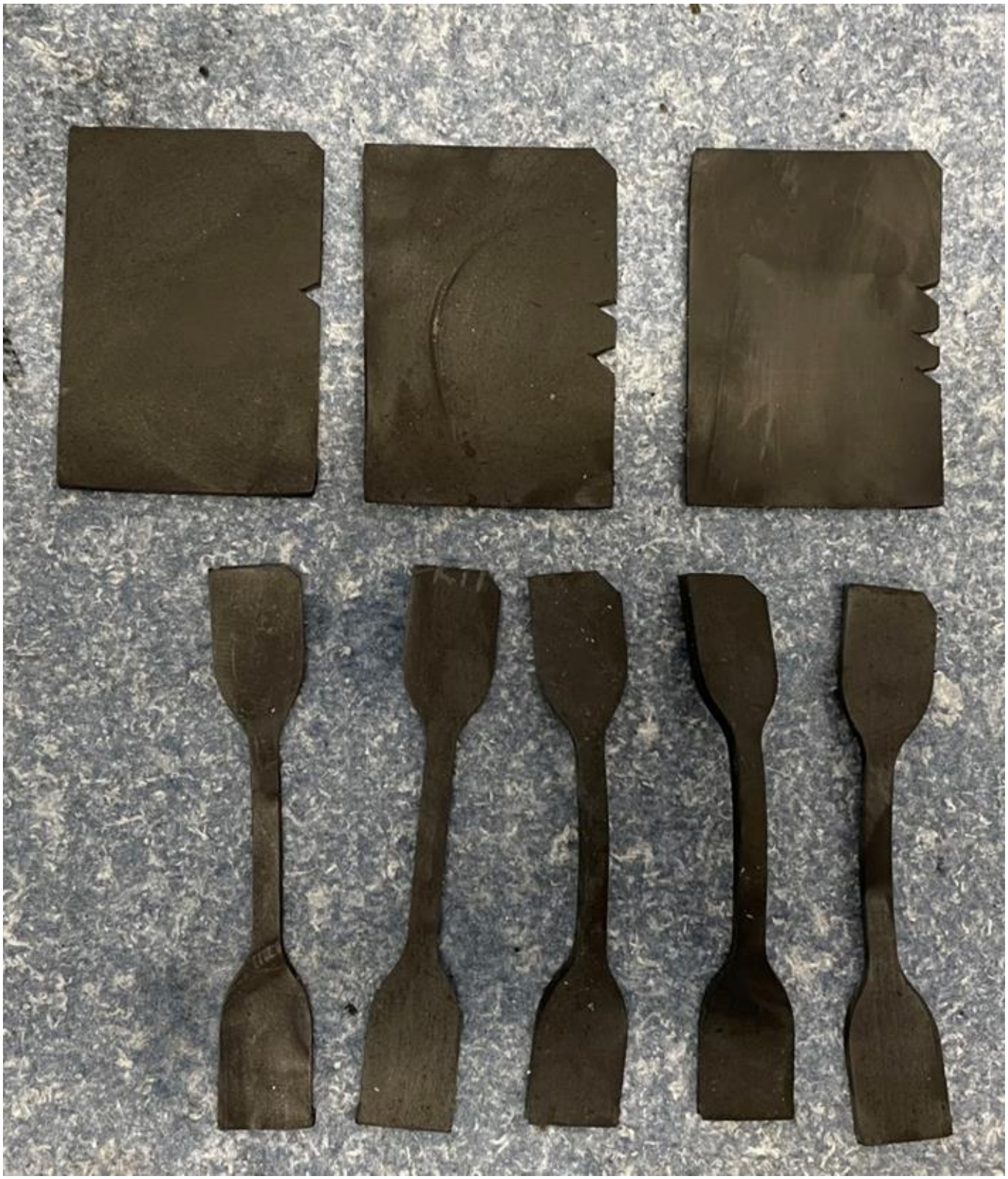


Figure D.5: KIFK11 after immersion

Figure D.6: KIFK15 after immersion

APPENDIX E Stress vs strain curve of all materials

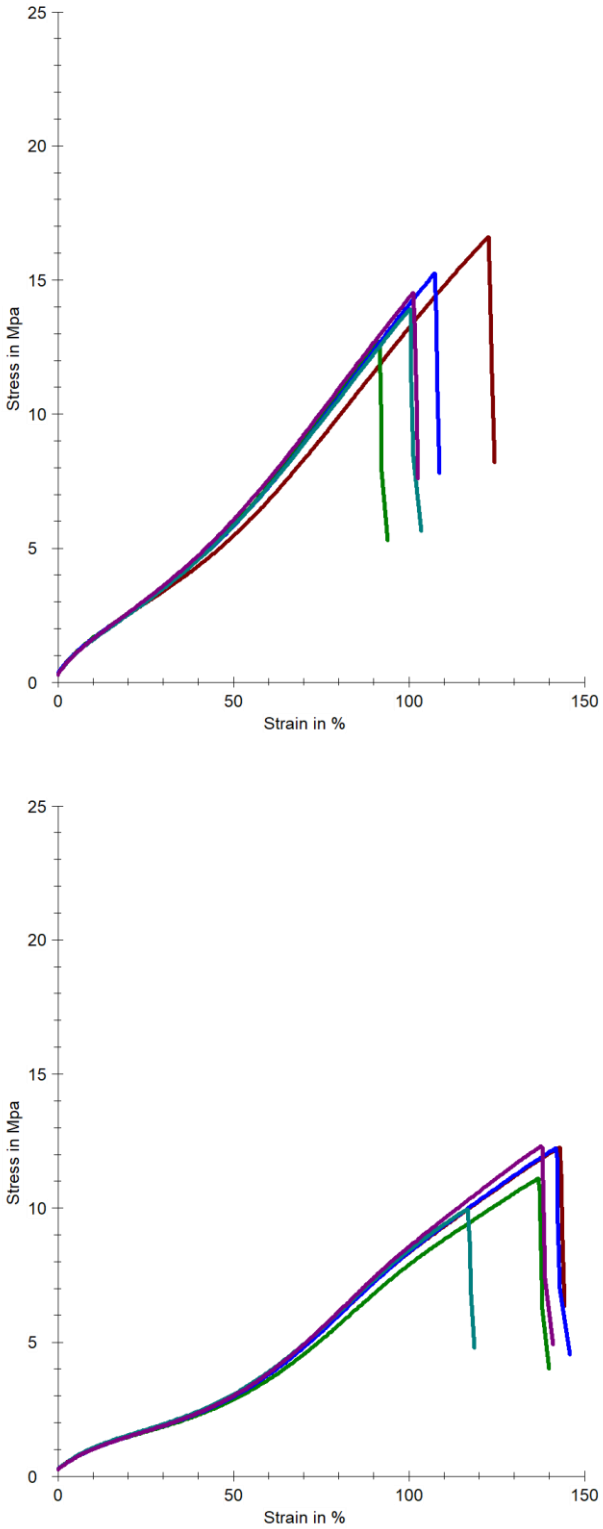


Figure E.1 Stress vs. strain curves of KIFK10 before (above) and after (below) immersion

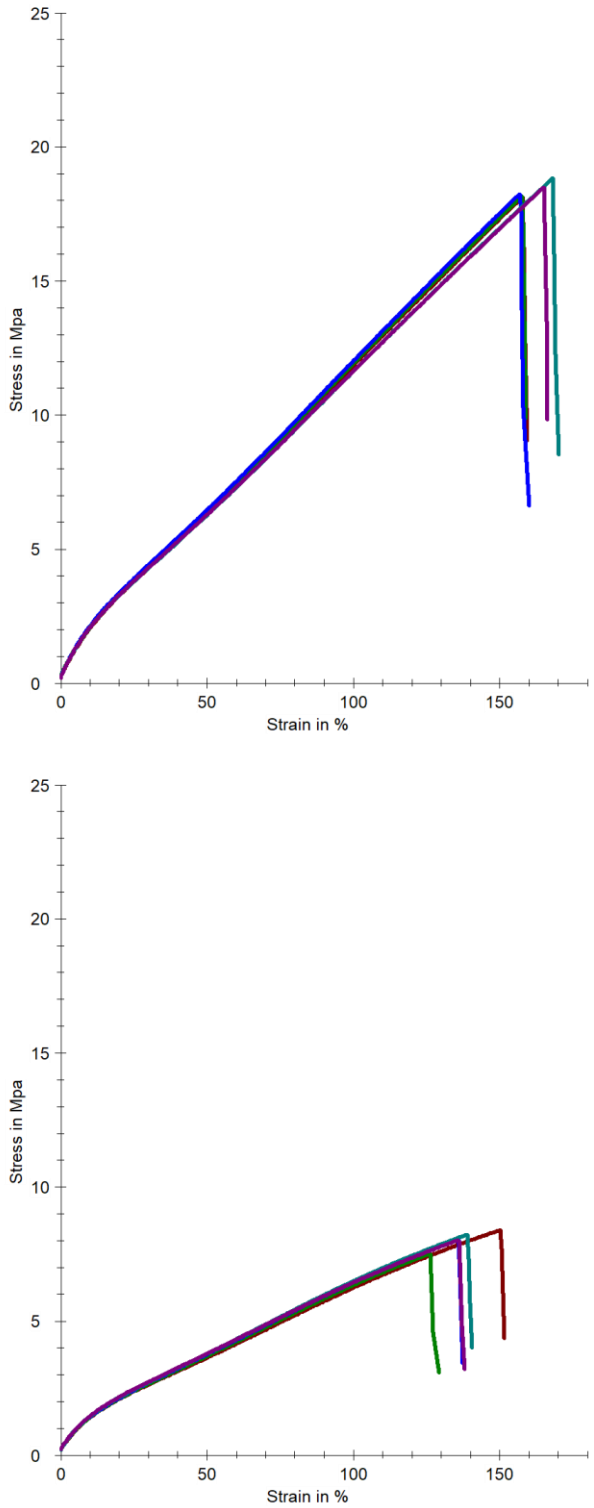


Figure E.2 Stress vs. strain curves of KIFK11 before (above) and after (below) immersion

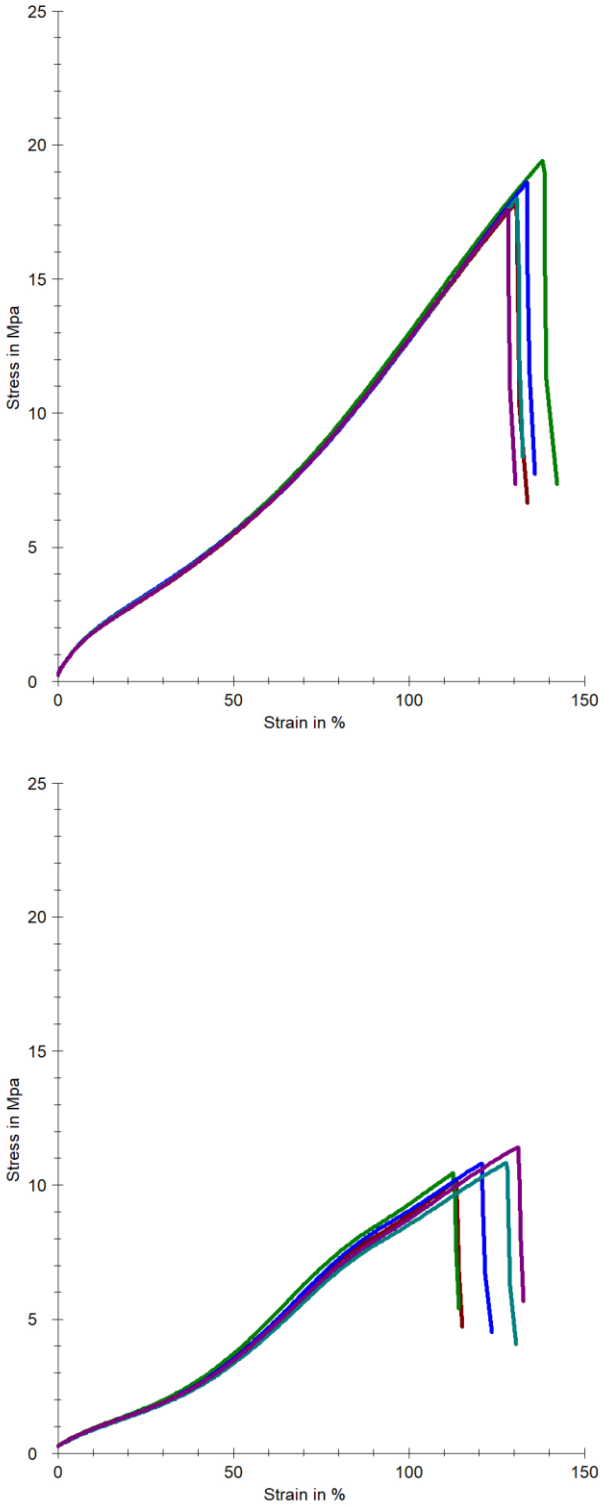


Figure E.3 Stress vs. strain curves of KIFK15 before (above) and after (below) immersion