

Supplementary information S5

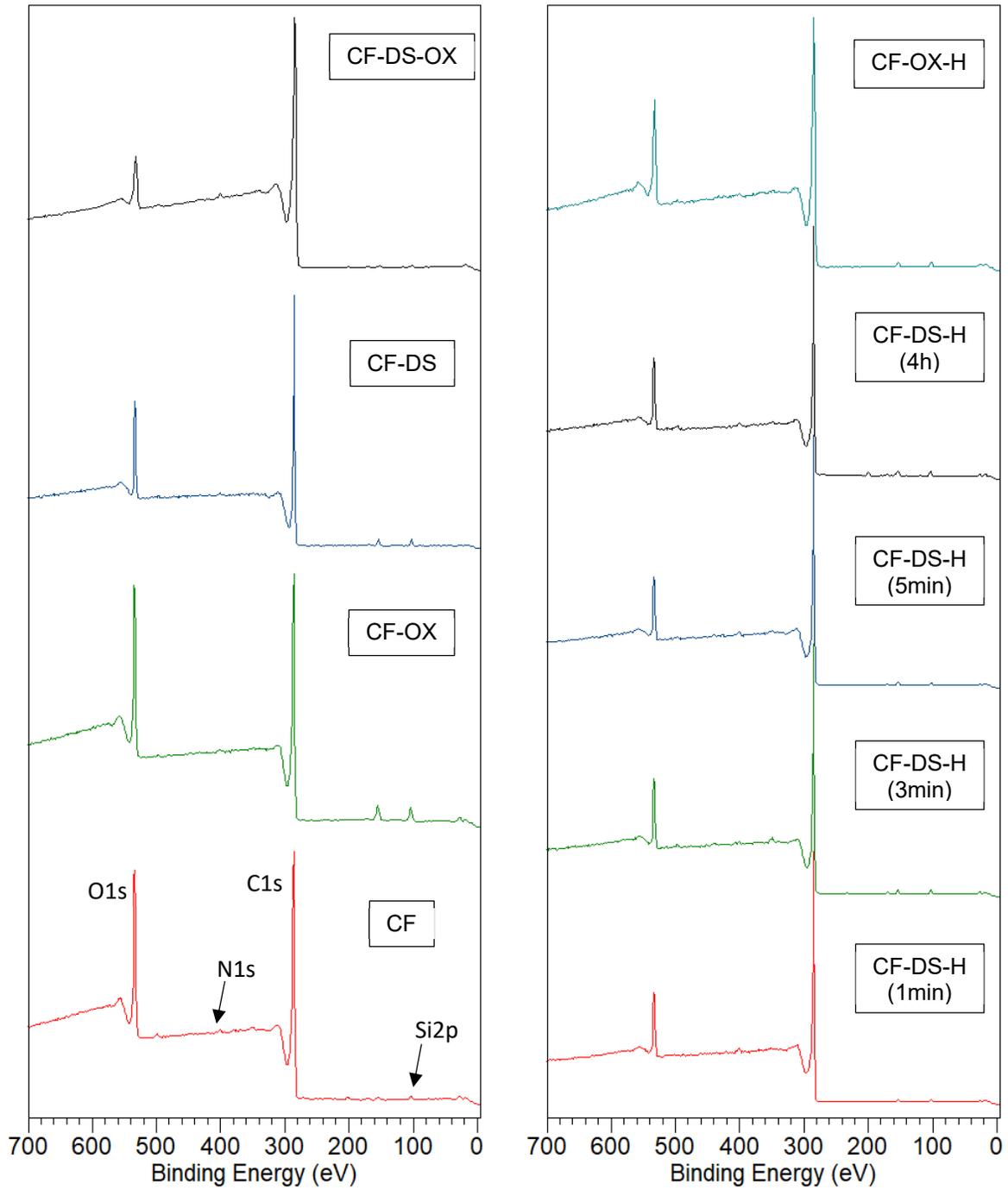


Figure 1. XPS survey for all samples, as indicated. The peaks considered are shown (C1s, O1s, N1s and Si2p)

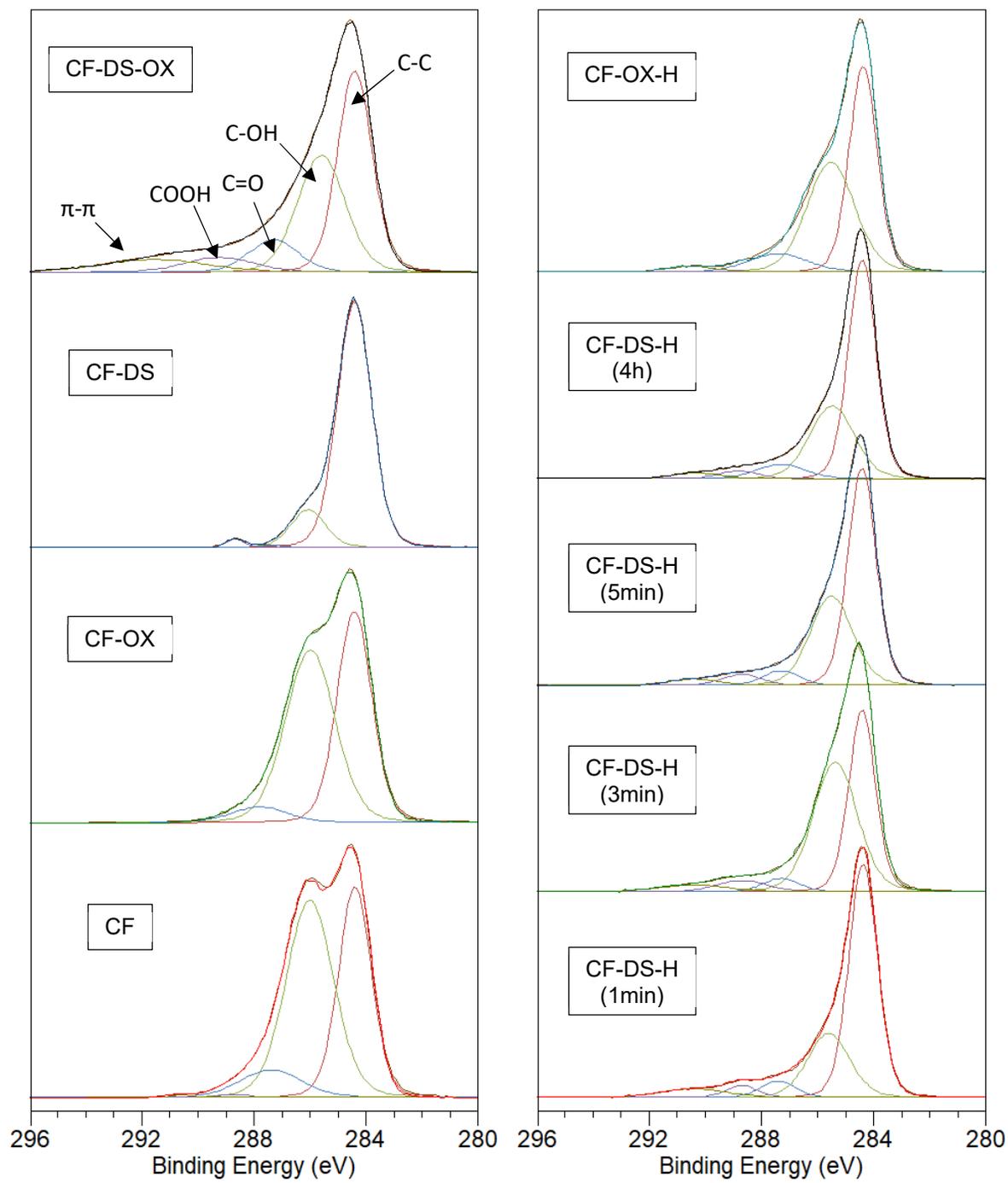


Figure 2. C1s peak deconvolution for all samples, as indicated.

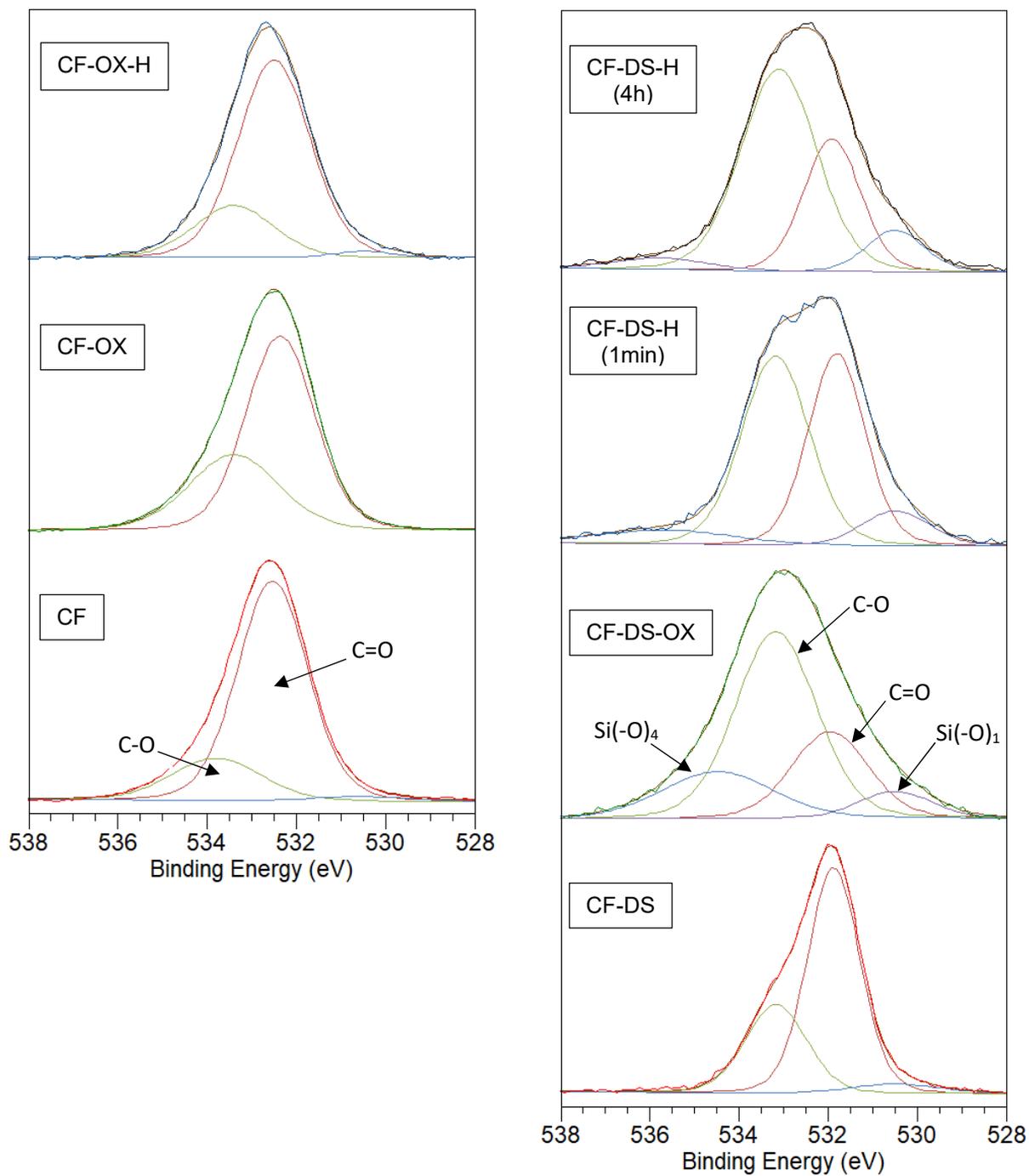


Figure 3. O1s peak deconvolution for samples indicated.

In Fig. 3 at left side we compare the samples without sizing removal. All lines fit with two peaks around 532.5 and 533.8 eV [1,2], corresponding to C=O and C-O. On CF and CF-OX-H, small

peaks around 530.5 eV became necessary, that may be correlated with Si(-O)₁ [3]. Without the sizing removal the main surface is the sizing polymer, in with C=O and C-O are broader than in samples shown at right side.

Samples show at right side of Fig. 3 are the ones with sizing removal before further pretreatment. Since CF-DS-H samples for 1, 3 and 5 min were similar, only the 1 min is shown. The corresponding C=O and C-O peaks are narrower, and their positions are around 531.8 and 533.2 eV [1,2]. The CF-DS sample already show the Si(-O)₁ peak around 530.5 eV. Either direct CF-DS oxidation (CF-DS-OX sample) or the samples treated by HMDSO have the O1s line much broader and asymmetrical. All then show an increase in the peak around 530.5 eV and one more peak in the range between 534.5 and 535.5 eV. On the CF-DS-OX sample, this last peak has higher intensity and may be correlated with the higher Si(-O)₄ content in the Si2p line [3]. From this analysis we may infer that the oxidation states shown in the Si2p line also appear in the O1s line, even though superimposed by the C=O and C-O peaks. The Si(-O)₂ and Si(-O)₃ oxidation states are perhaps indistinguishable from C=O and C-O peaks.

References

- [1] P.M.A. Sherwood, Surface analysis of carbon and carbon fibers for composites, *J. Electron Spectrosc. Relat. Phenom.* 81 (1996) 319–342. [https://doi.org/10.1016/0368-2048\(95\)02529-4](https://doi.org/10.1016/0368-2048(95)02529-4).
- [2] Y.-Q. Wang, F.-Q. Zhang, P.M.A. Sherwood, X-ray Photoelectron Spectroscopic Study of Carbon Fiber Surfaces. 23. Interfacial Interactions between Polyvinyl Alcohol and Carbon Fibers Electrochemically Oxidized in Nitric Acid Solution, *Chem. Mater.* 11 (1999) 2573–2583. <https://doi.org/10.1021/cm9902772>.
- [3] Y.-S. Lin, Y.-C. Lai, J.-H. Chen, C.-Y. Wu, Cold atmospheric pressure plasma-polymerized organosilicon oxide films for enhancing scratch resistance of flexible carbon fiber-reinforced polymer composites, *Thin Solid Films.* 686 (2019) 137420. <https://doi.org/10.1016/j.tsf.2019.137420>.

