1 实验目标

[1] 通过二分法不断迭代寻找合适的参数

1.1 目标参数

电压、喷射距离、滚筒转速、挤出速率、滑台移速、nZr：PVP 配比

需要注意的是，电压和喷射距离本质上是服务于同一个参数: 胶液飞行速度。这两个参数应当整体调节。

预想机理 通过控制胶液飞行速率避免胶液在飞行途中聚团，从而减少附着纤维中的聚团现象；挤出速率：避免在飞行速率过慢时挤出较多导致胶液束过粗；滑台移动速率：减少滑台变速抖动，抑制胶液偏聚；配比：控制纤维的分布；滚筒转速：尽快分离前后附着胶液的空间分布。

2 设备改良

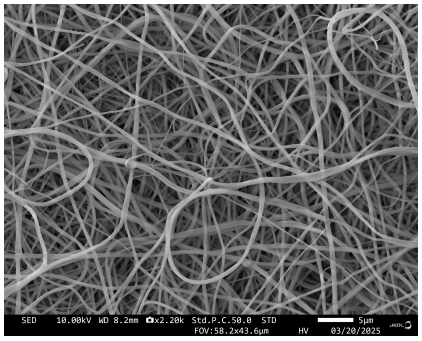
铝箔纸换硅油纸、提高箱体气密性

3 实验计划

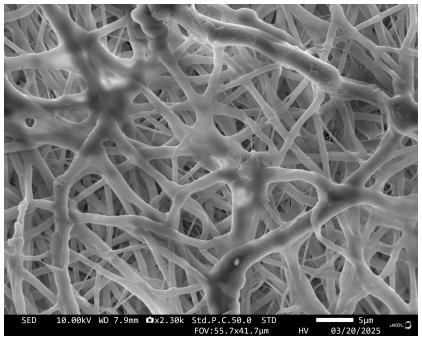
次序	参数	最初预设调参节点
1	nZr: PVP	1.5、2.5
2	滚筒转速	200+n40rpm
3	滑台移速	10mm/h

电压与喷射距离（喷射速率）的调节需要进行数学计算

4 0321 改良效果



(a) 标准参数：修正为 nZr：PVP=2：0.6

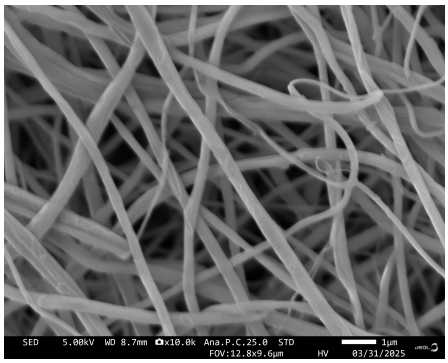


(b) 标准参数

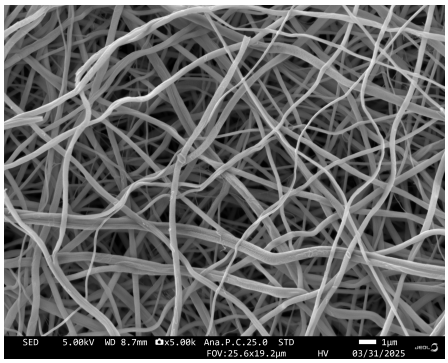
图 1a 可以发现此时纤维均匀度大幅提高，但纤维束与纤维束之间的直径差距开始显现明显，目测在十纳米和数百纳米之间波动，纤维束之间较之图 1b 更疏松。图 1b 可以发现此时纤维不均匀，单条纤维束上直径差距大，在 1 微米到 3 微米之间波动，而纤维束之间出现了粘连成块状的区域，多条纤维束之间存在并列粘连，较之图 1a 更密集，密度更高，预测密度较之图 1a 更低。

5 0401 效果

将图 2c 与图 1a 对比, 我们可以发现, 图 2c 的纤维相对不光滑, 对高温下力学性能来说可能会有影响。形成原因来看，不太可能是因为挤出速率更慢与 PvP：Zr 的含量配比，根据同行的经验来看，有可能是因为封箱的问题：封箱了箱子里湿度更低，失去了水汽的电场均匀化作用，导致胶液在飞行过程中精细拉伸较弱，因而形成的纤维束不太均匀。需要实现湿度的可控化。

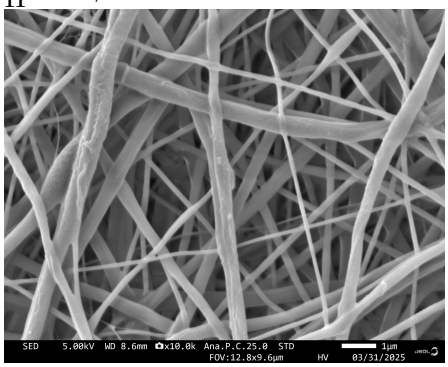


(a) nZr:PvP=0.64:1.725; 喷射速率 $1.8ml/h$

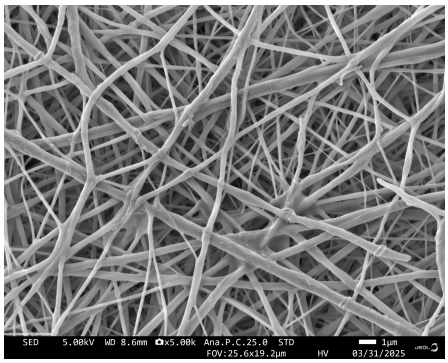


(b) nZr:PvP=0.64:1.725; 喷射速率 $1.5ml/h$

H

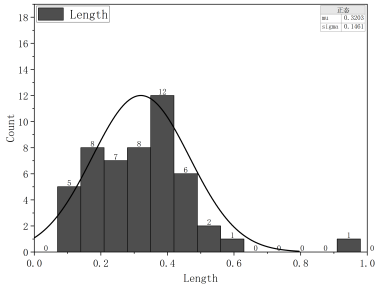


(c) nZr:PvP=0.66:2.75; 喷射速率 $1.5ml/h$

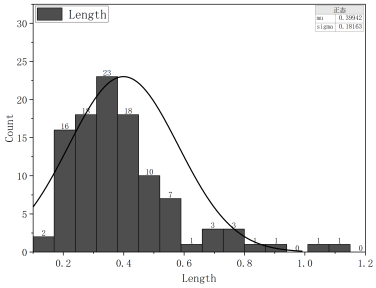


(d) nZr:PvP=0.66:2.75; 喷射速率 $1.8ml/h$

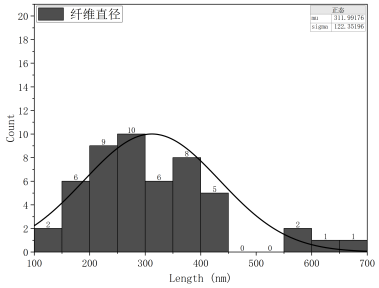
图 2: 三月三十一日四份样品 sem 图



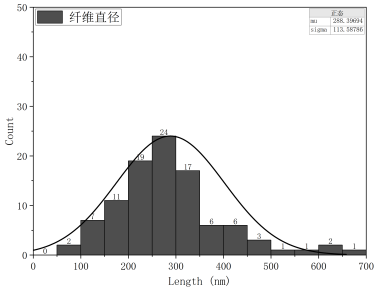
(a) 图 2c 纤维直径分布



(b) 图 2d 纤维直径分布



(c) 图 2a 纤维直径分布



(d) 图 2b 纤维直径分布

图 3: 图 2 纤维直径分布图

可以看到，1、2、4 的样品的 RL 非常不错，有继续研究的价值，当然导师希望我们先复现十多份二号样先好准备后续研究。

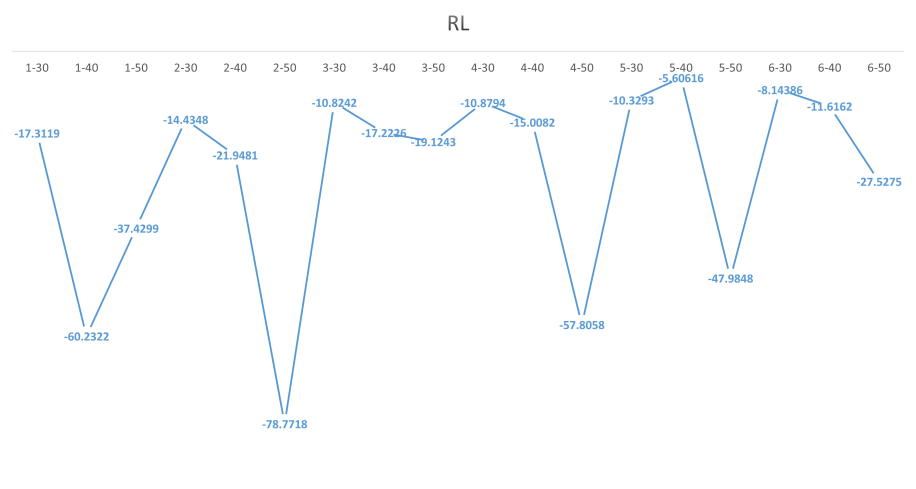


图 4: 四月一号样品 RL 系数

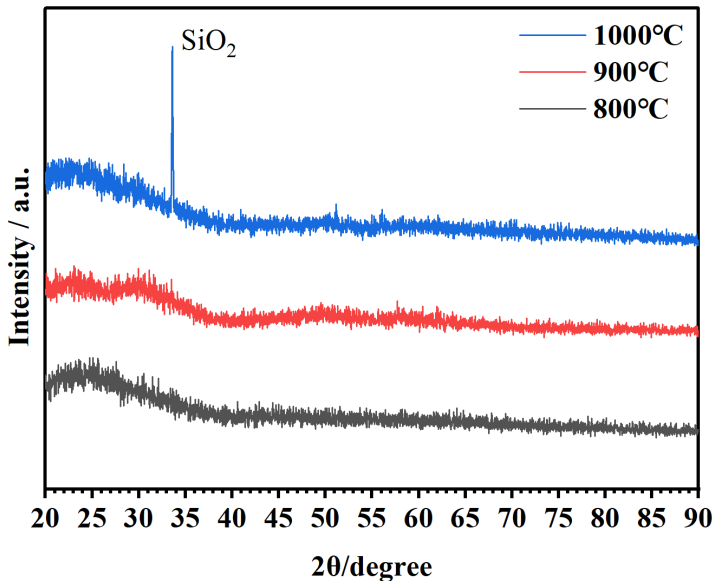
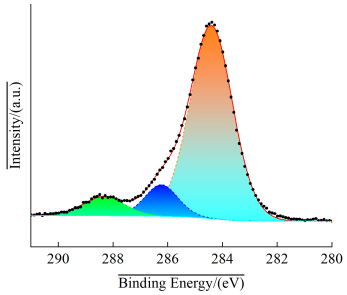
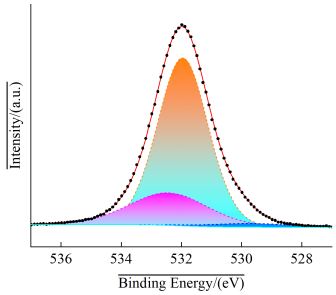


图 5: 从上至下 nZr=0.64/0.78/0.64

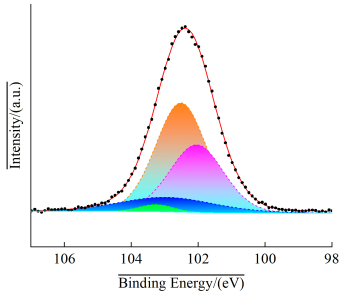
6 月 10 日回传 xps 数据一份：



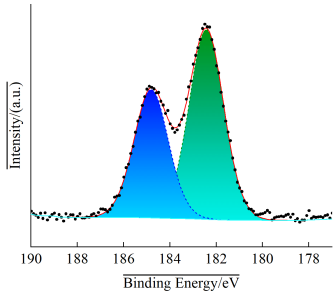
(a) nZr=0.78/PvP=1.725，800℃,C1s



(b) nZr=0.78/PvP=1.725,800℃，O1s

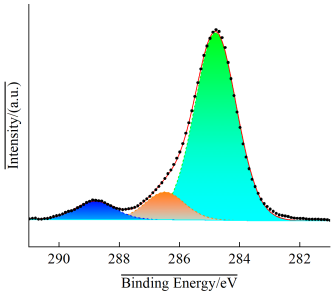


(c) nZr=0.78/PvP=1.725，800℃，Si2p

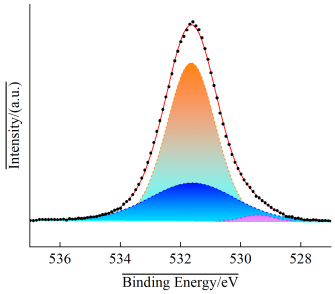


(d) nZr=0.78/PvP=1.725,800℃，Zr3d

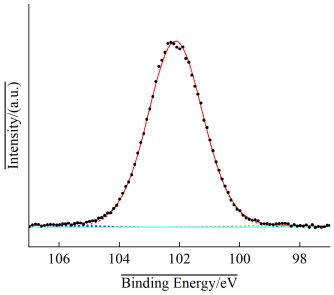
图 6: nZr=0.78,800℃ 组



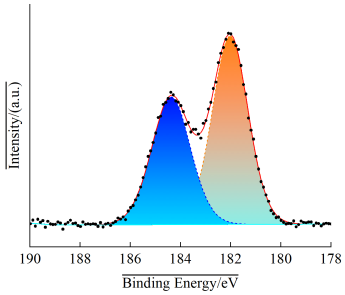
(a) nZr=0.78/PvP=1.725,900℃，C1s



(b) nZr=0.78/PvP=1.725,900℃，O1s

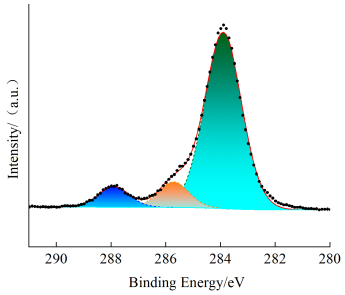


(c) nZr=0.78/PvP=1.725,900℃，Si2p

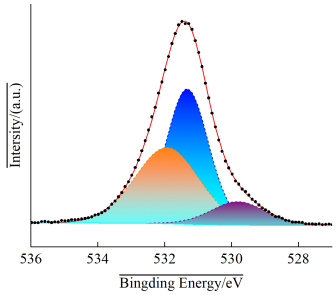


(d) nZr=0.78/PvP=1.725,900℃，Zr3d

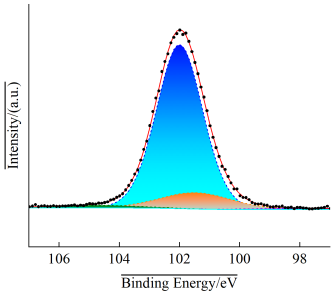
图 7: nZr=0.78,900℃ 组



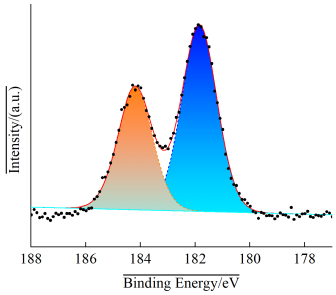
(a) nZr=0.64/PvP=1.725,1000℃，C1s



(b) nZr=0.64/PvP=1.725,1000℃，O1s



(c) nZr=0.64/PvP=1.725,1000℃，Si2p



(d) nZr=0.64/PvP=1.725,1000℃，Zr3d

图 8: nZr=0.64,1000℃ 组

图 6c 与图 7c 对比发现图 7c 并没有四个子峰，这是比较奇怪的一个事情，与此同时，图 8c 的子峰之间的结合能区别也不显著 e；

6 综述表

Material	Tickness (nm)	Minimum RL(dB)	EAB(GHz)	Ref.
Fe/SiC/PCS	2.25	-46.3	4.6(<-20dB)	[2]
MXene	2.7/2.1	-54.1(2.7)	7.76(2.1)	[3]
Fe/Co@C-CNFs	1.08/1.22	-18.66(1.08)	4.2(1.22)	[4]
PAN/Fe_3O_4	4	-11.3	7	[5]
TiN/carbon-paraffin composites	1.9	-41.8	3.9	[6]
SiC	1.9	-57.8	12	[6]
ZnO/Co	3.0/2.6	-68.4(3.0)	5.9(2.6)	[7]
P−CNF/Fe	4.1	-44.86	3.28	[8]
CeO_2/NC	2.5	-42.95	8.48	[9]
Zr/SiC	3.5	-48.6	3.2	[10]
ZrC	1.25/1.0	-25.77(1.25)	3.04(1.0)	[11]
ZrO_2/ZrC/ZrB_2	2.4	-21	8.64	[12]
ZrO_2/ZrB_2/C	4.0	-54	3.1	[13]
ZS-HNFA-x	2.4	-53.2	6.4	[14]
ZrO_2/CF−rGO	3.14	-62.99	8.19	[15]
ZrO_2/C	2.5	-36	7.3	[16]

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