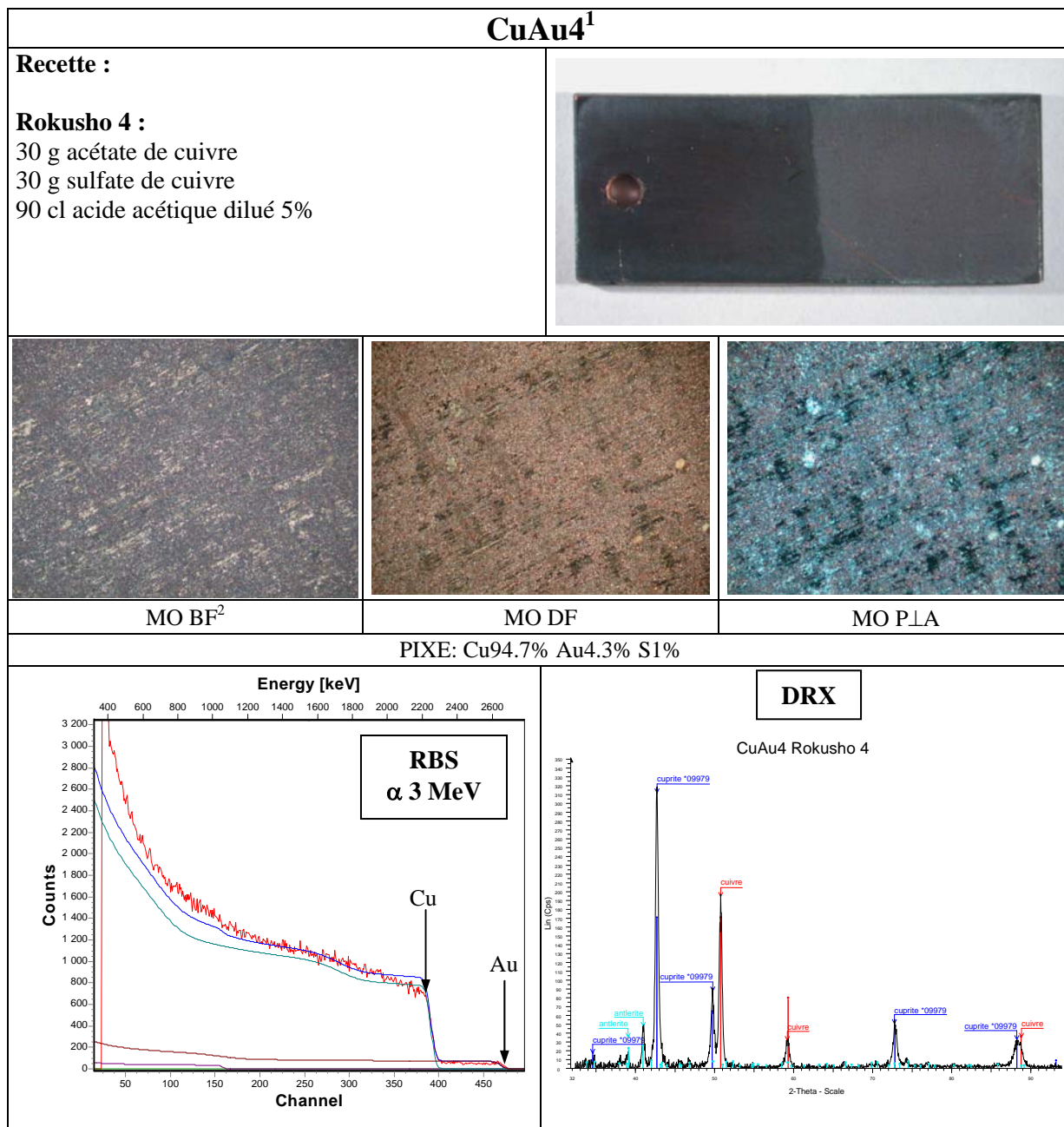


Annexe 7

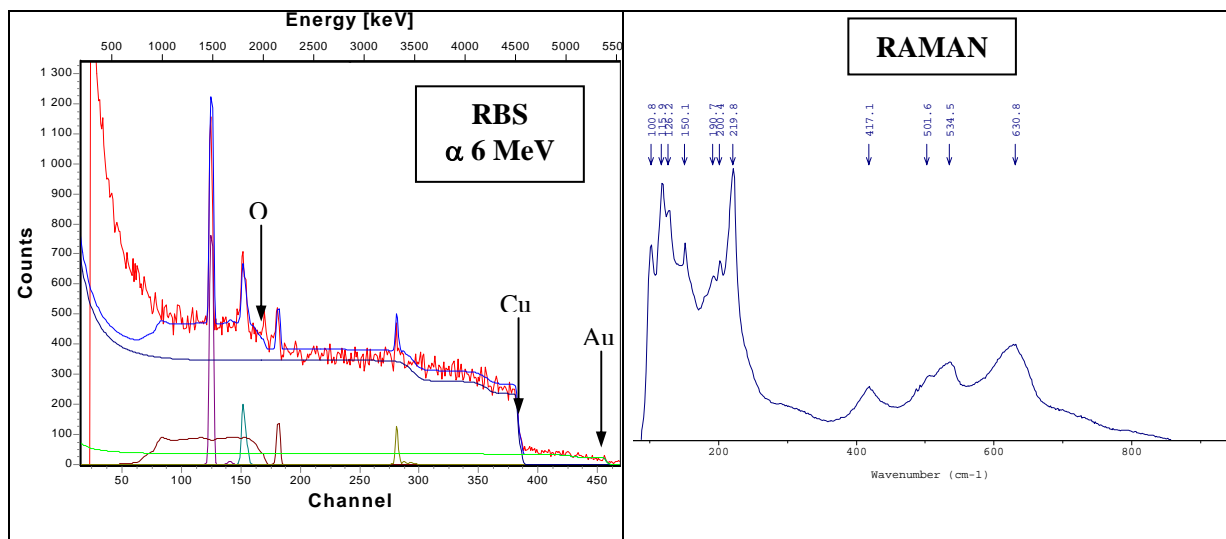
**Les patines expérimentales obtenues par
utilisation des recettes japonaises**

Résultats d'analyses



¹ La moitié des échantillons est vernie pour mettre en valeur la couleur de la couche de surface obtenue. Les analyses sont effectuées sur la partie non vernie.

² Le sigle MO signifie Microscopie Optique. BF indique que l'observation a été faite en fond clair (réflexion directe), DF qu'elle a été réalisée en fond noir (illumination oblique) et P⊥A qu'elle a été effectuée en lumière polarisée (polarisants croisés)



Description

Composition Couche			Diffraction X																																																																
Cu40 O25 Au0.5 Porosité +rugosité Cu/Au=80	7000.10 ¹⁵ At/cm ²		Cuprite décalée *0.9979																																																																
Cu66 Au1 O33 Cu/Au=66	9000.10 ¹⁵ At/cm ²		Cuivre non décalé																																																																
Cu98.4 Au1.3 Cu/Au=75.7	Substrat		Dépouillement RAMAN : cuprite seule, avec reconstruction des bandes complexes																																																																
Analyse XPS: Or métallique détecté : Signal Au 4f7/2 détecté à 84,45 eV soit 0,45 eV de plus que la valeur théorique de Au ⁰ 4f 7/2 (Au ³⁺ 4f 7/2 : 86 eV)			CuAu4 R4 - cm⁻¹																																																																
			<table border="1"> <thead> <tr> <th>sommet</th> <th>Amplitude</th> <th>largeur</th> <th>Surface</th> </tr> </thead> <tbody> <tr><td>100,51</td><td>4704,43</td><td>6,17</td><td>22233,9</td></tr> <tr><td>11,65</td><td>8416,24</td><td>13,29</td><td>102392</td></tr> <tr><td>128,04</td><td>5267,3</td><td>8,22</td><td>40656,7</td></tr> <tr><td>150,3</td><td>4476,77</td><td>7,95</td><td>33774</td></tr> <tr><td>178,38</td><td>744,91</td><td>1,52</td><td>1003,76</td></tr> <tr><td>190,3</td><td>1489,52</td><td>7,03</td><td>10051</td></tr> <tr><td>200,63</td><td>2806,78</td><td>8,48</td><td>22849,1</td></tr> <tr><td>219,74</td><td>7875,99</td><td>9,34</td><td>59231,6</td></tr> <tr><td>313,61</td><td>127,21</td><td>25,88</td><td>2585,03</td></tr> <tr><td>416,93</td><td>1285,65</td><td>24,88</td><td>25420,7</td></tr> <tr><td>483,12</td><td>262,81</td><td>9,91</td><td>2086,24</td></tr> <tr><td>504,08</td><td>1170,61</td><td>22,75</td><td>21388,8</td></tr> <tr><td>531,63</td><td>1661,03</td><td>20,04</td><td>26827</td></tr> <tr><td>586,76</td><td>424,51</td><td>28,04</td><td>9660,55</td></tr> <tr><td>627,55</td><td>2733,32</td><td>35,64</td><td>79453,2</td></tr> </tbody> </table>	sommet	Amplitude	largeur	Surface	100,51	4704,43	6,17	22233,9	11,65	8416,24	13,29	102392	128,04	5267,3	8,22	40656,7	150,3	4476,77	7,95	33774	178,38	744,91	1,52	1003,76	190,3	1489,52	7,03	10051	200,63	2806,78	8,48	22849,1	219,74	7875,99	9,34	59231,6	313,61	127,21	25,88	2585,03	416,93	1285,65	24,88	25420,7	483,12	262,81	9,91	2086,24	504,08	1170,61	22,75	21388,8	531,63	1661,03	20,04	26827	586,76	424,51	28,04	9660,55	627,55	2733,32	35,64	79453,2
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CuAg1Au3

Recette :

Rokusho 4 :

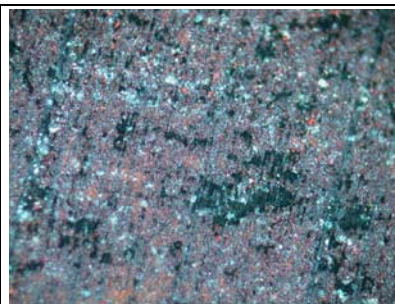
- 30 g acétate de cuivre
- 30 g sulfate de cuivre
- 90 cl acide acétique dilué 5%



MO BF

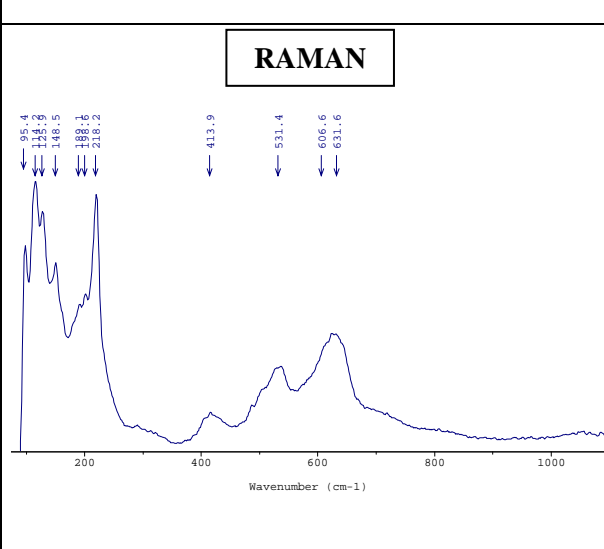
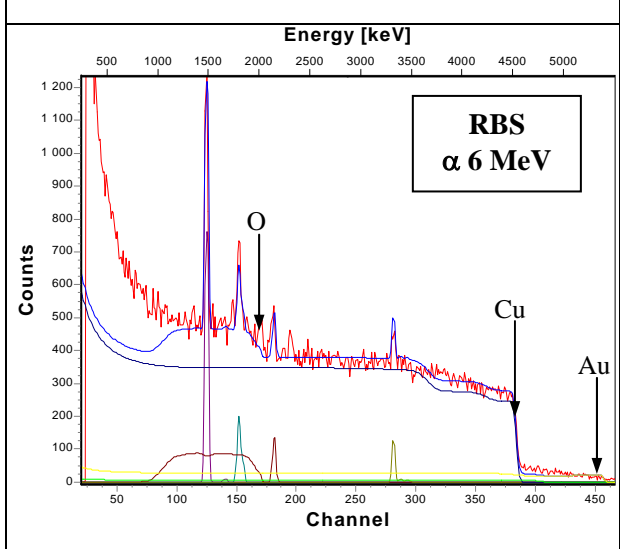
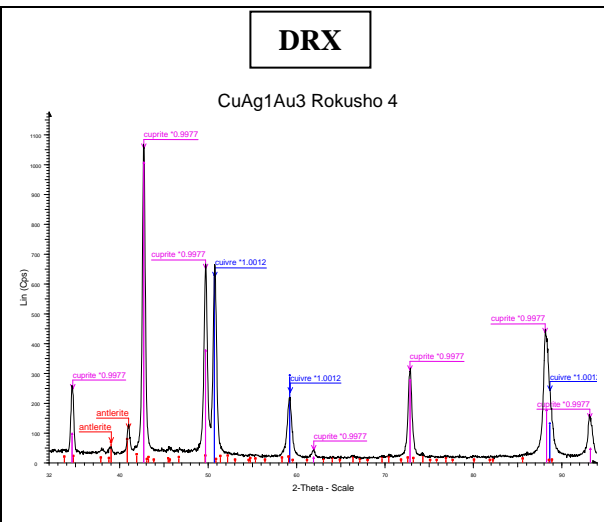
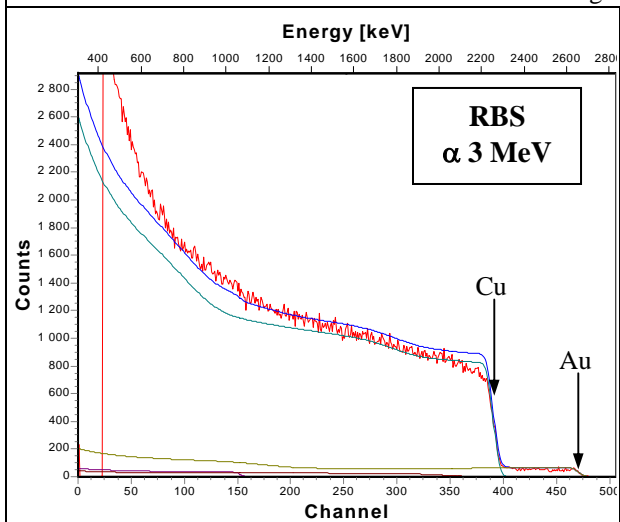


MO DF



MO PL

PIXE: Cu95% Ag1.1% Au3.1% S0.8%



Description			
Composition Couche			
Cu45 O25 Au0.5 Porosité + rugosité Cu/Au=90	6000.10 ¹⁵ At/cm ²	Diffraction X	
		Cuprite Décalée *0.9977	
Cu66 Ag0.6 Au0.6 O33 Cu/Au=110 Cu/Ag=110	8000.10 ¹⁵ At/cm ²	Cuivre Décalé *1.0012	
Cu98.4 Au1 Ag0.6 Cu/Au=98.4 Cu/Ag=164	Substrat	Dépouillement RAMAN : cuprite seule, avec reconstruction des bandes complexes	
		CuAg1Au3 R4 - cm⁻¹	
		sommet	Amplitude
		largeur	Surface
		95,59	725,18
		5,49	3296,95
		113,12	1511,37
		13,49	17671,7
		127,32	1073,32
		13,14	13059,7
		148,89	611,54
		13,05	6146,07
		178,91	96,54
		5,81	433,73
		189,61	243,23
		9,33	1755,77
		199,79	356,82
		9,68	2978,96
		217,78	1458,18
		11,81	14204,6
		412,95	213,65
		29,92	5078,47
		485,11	111,08
		8,64	879,85
		502,33	185,12
		14,38	2137,95
		527,76	372,47
		23,25	6976,9
		599,1	664,51
		29,04	15733,1
		625,34	223,25
		28,56	5181,13
		647,97	285,39
		19,68	4592,4

CuSn3Ag2Au6

Recette :

Rokusho 4 :

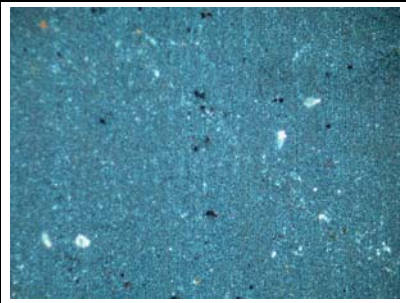
- 30 g acétate de cuivre
- 30 g sulfate de cuivre
- 90 cl acide acétique dilué 5%



MO BF

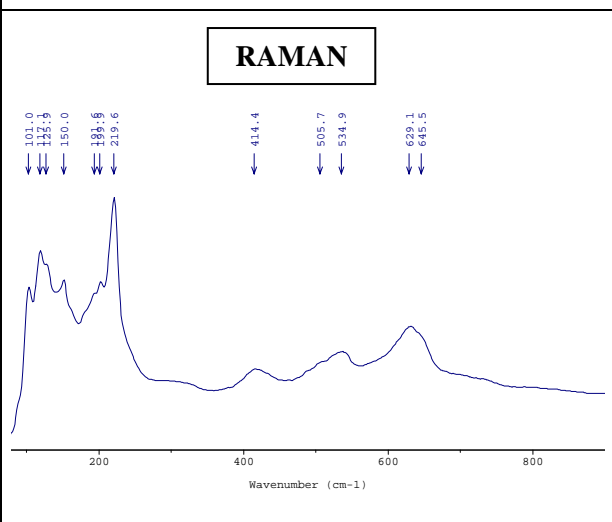
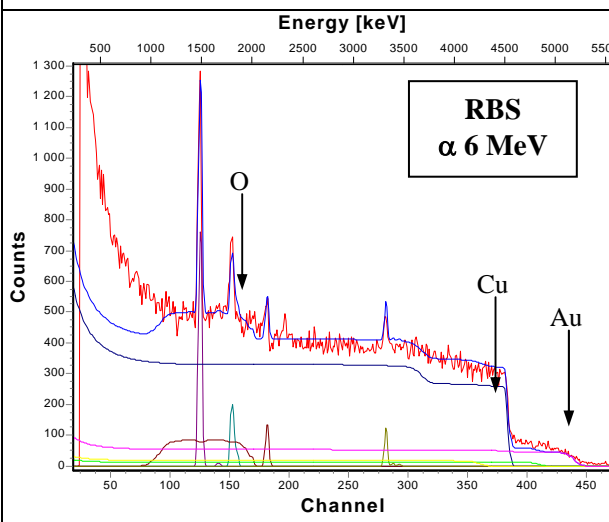
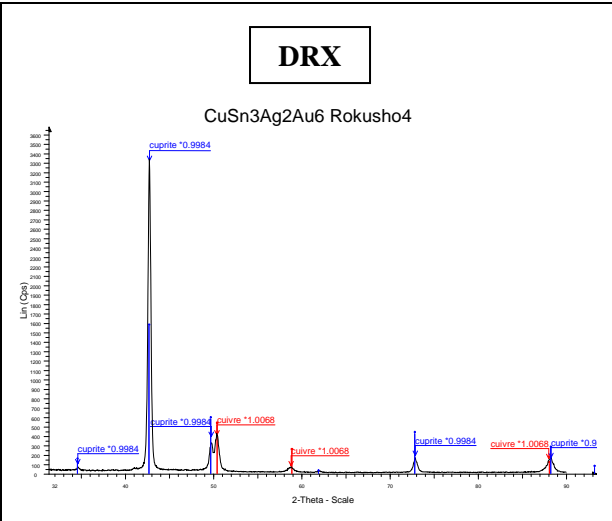
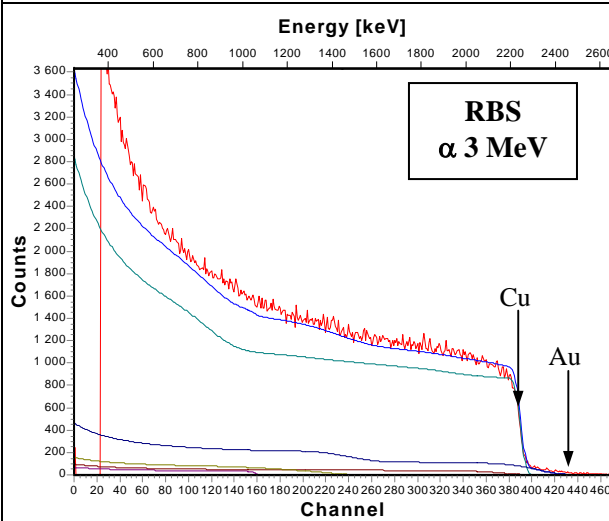


MO DF



MO PLA

PIXE: Cu87.5% Sn3.3% Ag2.6% Au6.3% S0.3%



Description																																																																							
Composition Couche																																																																							
Cu50 O25 Ag0 Au0 Porosité +rugosité	4000.10 ¹⁵ At/cm ²	Diffraction X Cuprite Décalée *0.9984 Cuivre Décalé *1.0068 Dépouillement RAMAN : cuprite seule, avec reconstruction des bandes complexes CuSn3Ag2Au6 R4 cm ⁻¹ <table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th>sommet</th> <th>Amplitude</th> <th>largeur</th> <th>Surface</th> </tr> </thead> <tbody> <tr><td>100,82</td><td>2169,83</td><td>9,37</td><td>16247,5</td></tr> <tr><td>116,23</td><td>2482,35</td><td>10,7</td><td>22522</td></tr> <tr><td>126,96</td><td>2595,76</td><td>19,93</td><td>40600,6</td></tr> <tr><td>149,91</td><td>1872,58</td><td>13,27</td><td>19378,7</td></tr> <tr><td>180,35</td><td>220,88</td><td>5,69</td><td>973,29</td></tr> <tr><td>190,52</td><td>634,63</td><td>7,8</td><td>3829,69</td></tr> <tr><td>200,69</td><td>1396,92</td><td>12,28</td><td>15537,1</td></tr> <tr><td>219,46</td><td>5076,62</td><td>12,04</td><td>49019,6</td></tr> <tr><td>311,91</td><td>113,34</td><td>25,47</td><td>2265,6</td></tr> <tr><td>415,24</td><td>656,29</td><td>28,12</td><td>14598,3</td></tr> <tr><td>486,05</td><td>165,34</td><td>8,03</td><td>1064,53</td></tr> <tr><td>504,79</td><td>466,19</td><td>19,58</td><td>7333,75</td></tr> <tr><td>531,64</td><td>844,82</td><td>22,3</td><td>15183,2</td></tr> <tr><td>599,98</td><td>302,92</td><td>23,28</td><td>5714,49</td></tr> <tr><td>629,99</td><td>1750,44</td><td>30,91</td><td>44257,6</td></tr> <tr><td>650,13</td><td>386,71</td><td>17,16</td><td>5424,65</td></tr> </tbody> </table>		sommet	Amplitude	largeur	Surface	100,82	2169,83	9,37	16247,5	116,23	2482,35	10,7	22522	126,96	2595,76	19,93	40600,6	149,91	1872,58	13,27	19378,7	180,35	220,88	5,69	973,29	190,52	634,63	7,8	3829,69	200,69	1396,92	12,28	15537,1	219,46	5076,62	12,04	49019,6	311,91	113,34	25,47	2265,6	415,24	656,29	28,12	14598,3	486,05	165,34	8,03	1064,53	504,79	466,19	19,58	7333,75	531,64	844,82	22,3	15183,2	599,98	302,92	23,28	5714,49	629,99	1750,44	30,91	44257,6	650,13	386,71	17,16	5424,65
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650,13	386,71	17,16	5424,65																																																																				
Cu65 Ag 1 Au1 O33 Cu/Au=65 Cu/Ag=65	7000.10 ¹⁵ At/cm ²																																																																						
Cu64 Sn1 Ag 1 Au1.8 O32.2 Cu/Au=35.5 Cu/Ag=64	2000.10 ¹⁵ At/cm ²																																																																						
Cu94.5 Ag1.2 Au2 Sn1.7 Cu/Au=315 Cu/Ag=52	Substrat																																																																						
Analyse XPS: Or et Argent non visibles en surface ni même après une abrasion de 40 min (env. 400nm)																																																																							

CuSn6Ag3Au1

Recette :

Rokusho 4 :

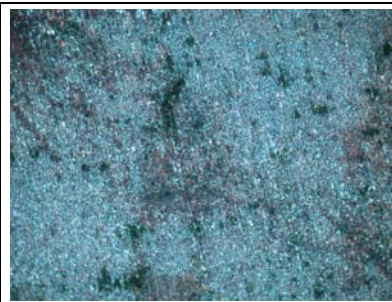
- 30 g acétate de cuivre
- 30 g sulfate de cuivre
- 90 cl acide acétique dilué 5%



MO BF

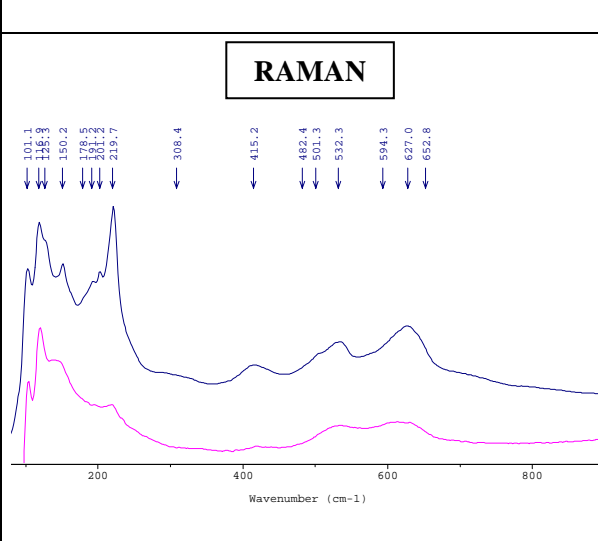
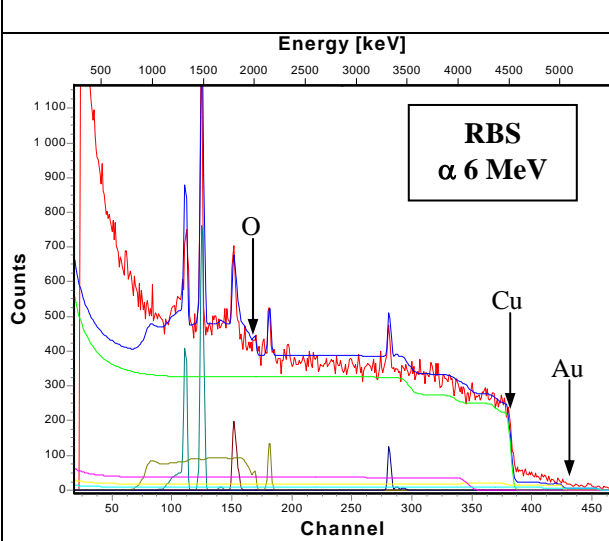
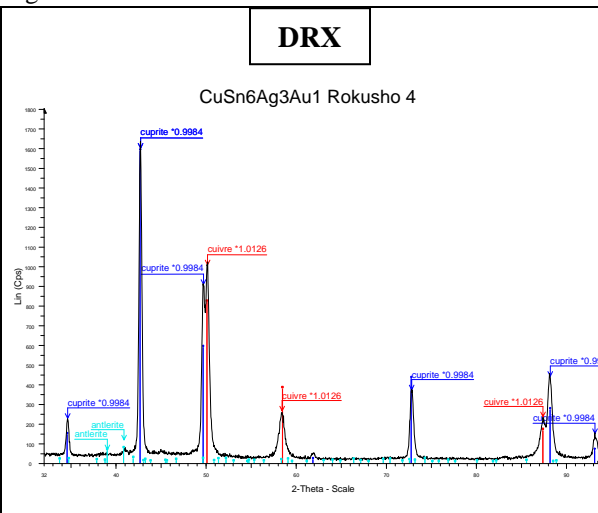
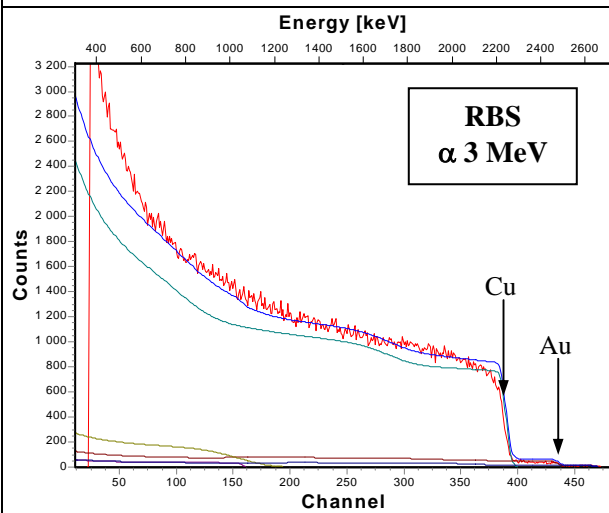


MO DF



MO PLA

PIXE: Cu88.7% Sn6.1% Ag3.6% Au1.1% S0.3%



tel-00011255, version 1 - 22 Dec 2005

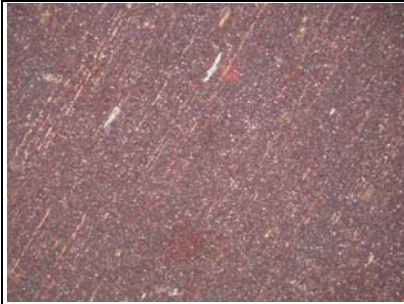
Description																																																																		
Composition Couche																																																																		
Cu40 O25 Ag 1 Au0.1 Porosité +rugosité Cu/Au=400 Cu/Ag=40	7000.10 ¹⁵ At/cm ²	Diffraction X																																																																
Cu66 Ag 1.8 Au0.3 O31.9 Cu/Au=220 Cu/Ag=36.6	7500.10 ¹⁵ At/cm ²	Cuprite Décalée *0.9984																																																																
Cu94.5 Ag1.8 Au0.3 Sn 3.4 Cu/Au=315 Cu/Ag=52	Substrat	Cuivre Décalé *1.0126																																																																
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sommet	Amplitude	largeur	Surface																																																															
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sommet	Amplitude	largeur	Surface																																																															
101,85	2139,92	4,02	6588,66																																																															
118,54	4754,04	11,34	44415,7																																																															
141,1	3155,17	28,51	69222,9																																																															
218,26	451,17	8,79	3080,47																																																															
528,85	1232,42	50,73	50380																																																															
611,5	1499,5	70,18	85659,4																																																															
1262,39	415,88	50,99	18786,3																																																															
1289,94	561,42	49,49	24702,8																																																															
1357,09	1743,39	78,42	122669																																																															

CuSn5Pb5

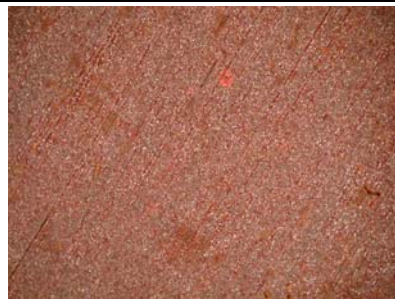
Recette :

Rokusho 4 :

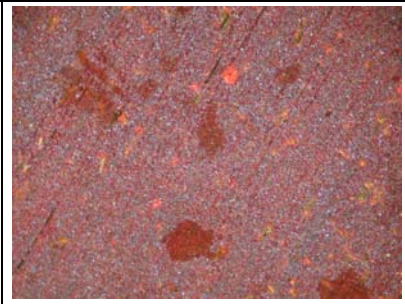
30 g acétate de cuivre
 30 g sulfate de cuivre
 90cl acide acétique dilué 5%



MO BF

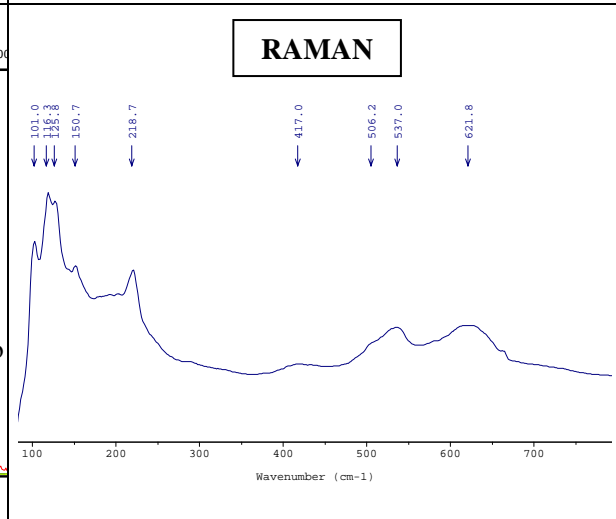
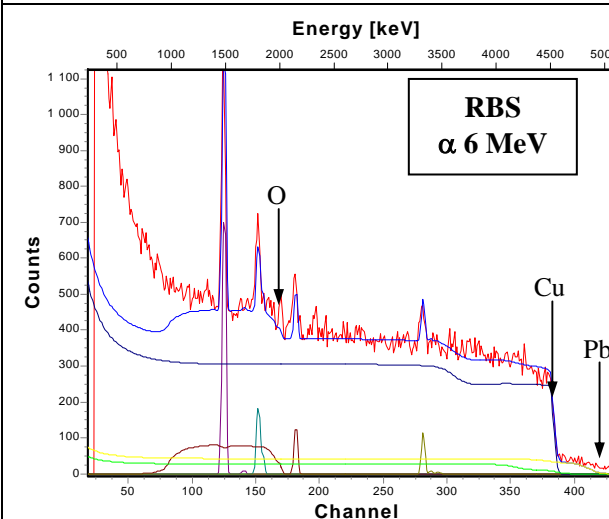
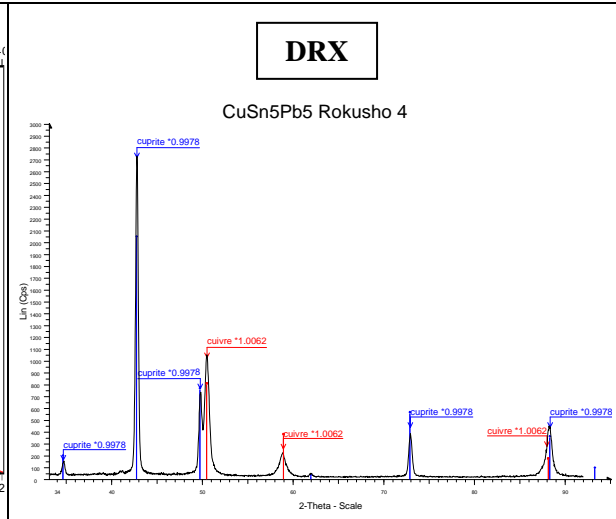
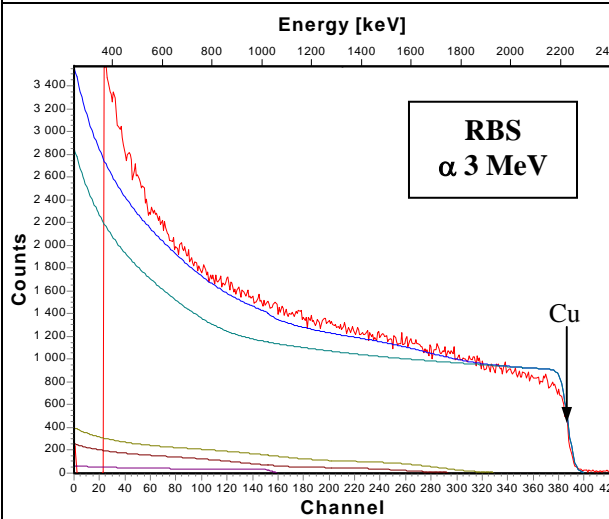


MO DF



MO P.L.A

PIXE: Cu94.4% Sn4.6% Pb0.9% S0.1%



Description

Composition Couche																																																																	
Cu60 O30 Porosité +rugosité	9000.10 ¹⁵ At/cm ²	Diffraction X Cuprite Décalée *0.9978 Cuivre Décalé *1.0062 Dépouillement RAMAN : cuprite seule, avec reconstruction des bandes complexes CuSn5Pb5 R4 cm ⁻¹ <table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th>sommet</th> <th>Amplitude</th> <th>largeur</th> <th>Surface</th> </tr> </thead> <tbody> <tr><td>101,18</td><td>1483,45</td><td>3,86</td><td>4385,51</td></tr> <tr><td>117,27</td><td>3558,9</td><td>9,52</td><td>30758,6</td></tr> <tr><td>125,82</td><td>3963,29</td><td>13,79</td><td>47625,9</td></tr> <tr><td>150,19</td><td>110,34</td><td>10,99</td><td>9400,9</td></tr> <tr><td>190,65</td><td>127,46</td><td>7,05</td><td>695,43</td></tr> <tr><td>201,21</td><td>230,04</td><td>10,68</td><td>2351,46</td></tr> <tr><td>219,3</td><td>1571,92</td><td>10,17</td><td>12415,3</td></tr> <tr><td>416,96</td><td>337,46</td><td>28,64</td><td>7683,15</td></tr> <tr><td>485,85</td><td>126,75</td><td>18,13</td><td>1842,12</td></tr> <tr><td>508,23</td><td>810,62</td><td>19,92</td><td>12974</td></tr> <tr><td>532,34</td><td>1489,1</td><td>21,77</td><td>26131,9</td></tr> <tr><td>585,73</td><td>230,93</td><td>23,63</td><td>4427,64</td></tr> <tr><td>621,9</td><td>1520,48</td><td>35,53</td><td>44026,2</td></tr> <tr><td>643</td><td>173,01</td><td>11,28</td><td>1594,76</td></tr> </tbody> </table>				sommet	Amplitude	largeur	Surface	101,18	1483,45	3,86	4385,51	117,27	3558,9	9,52	30758,6	125,82	3963,29	13,79	47625,9	150,19	110,34	10,99	9400,9	190,65	127,46	7,05	695,43	201,21	230,04	10,68	2351,46	219,3	1571,92	10,17	12415,3	416,96	337,46	28,64	7683,15	485,85	126,75	18,13	1842,12	508,23	810,62	19,92	12974	532,34	1489,1	21,77	26131,9	585,73	230,93	23,63	4427,64	621,9	1520,48	35,53	44026,2	643	173,01	11,28	1594,76
sommet	Amplitude					largeur	Surface																																																										
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Cu67.2 Sn1 Pb0.8 O31	5000.10 ¹⁵ At/cm ²																																																																
Cu95.6 Sn2.8 Pb1.6 Cu/Au=315 Cu/Ag=52	Substrat																																																																

CuSn3Ag2Au6

Recette :

Murakami :

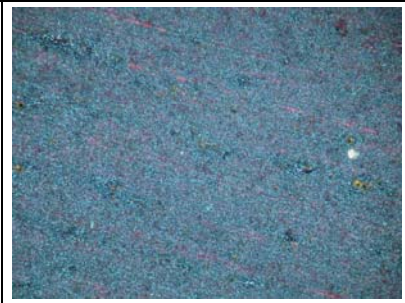
- 1,9 g acétate de cuivre
- 1,2 g sulfate de cuivre
- 0,1 g alun
- 1 l eau



MO BF

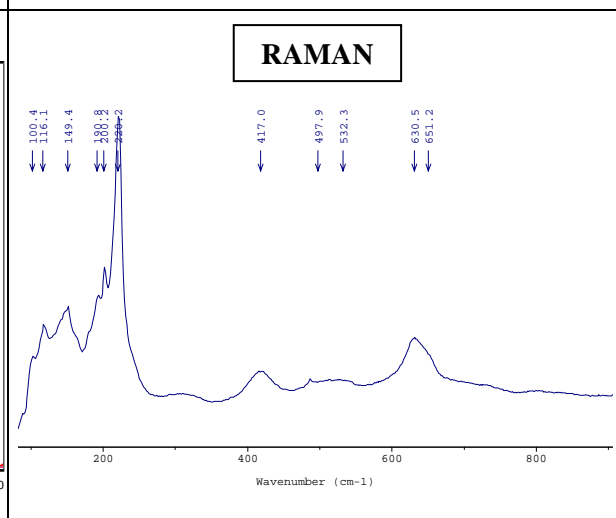
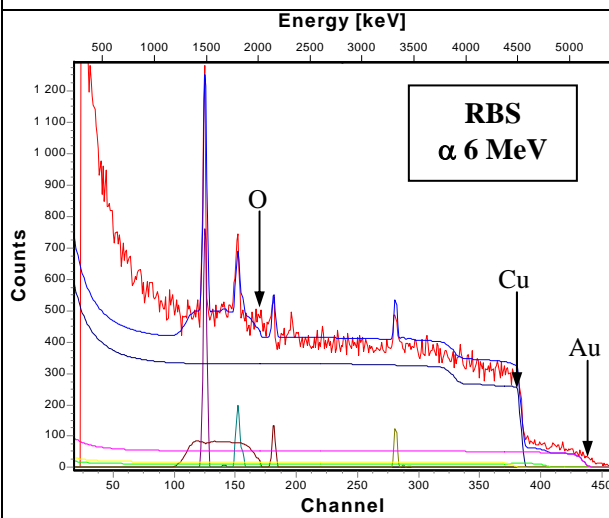
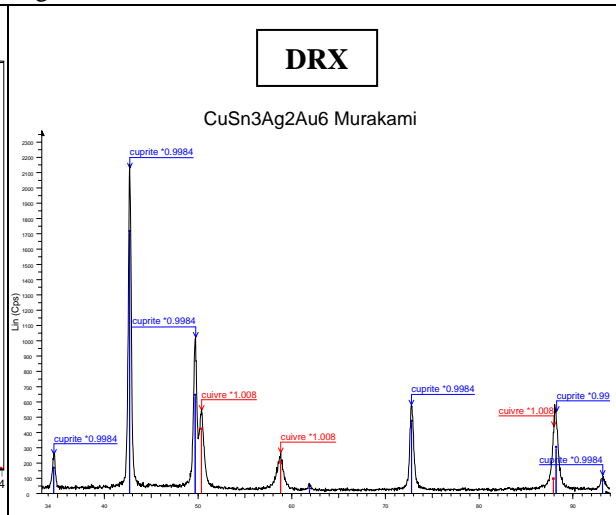
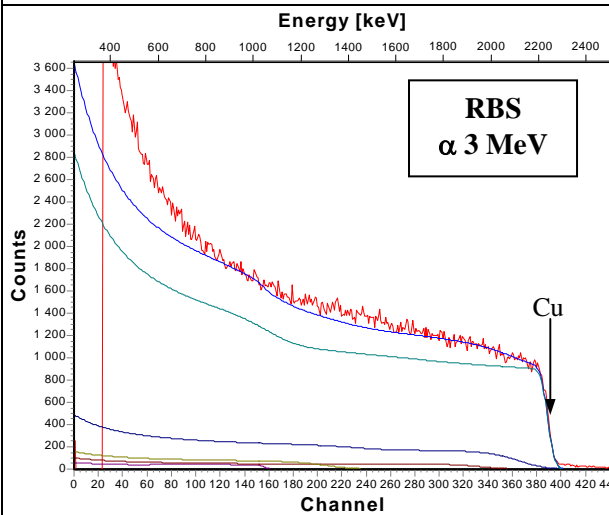


MO DF



MO P.LA

PIXE: Cu89.1% Ag2.4% Au5.7%



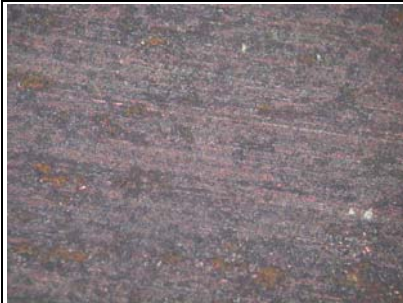
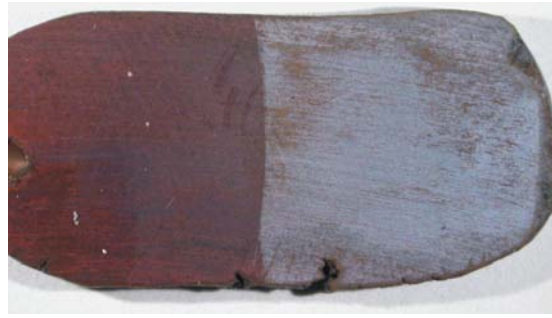
Description		
Composition Couche		
Cu63 O37 Ag0 Au0 +rugosité	5000.10 ¹⁵ At/cm ²	Diffraction X
Cu68 Ag1.2 Au1.5 O29 Cu/Au=45 Cu/Ag=57	5500.10 ¹⁵ At/cm ²	Cuprite Décalée *0.9984
Cu94.5 Ag1.2 Au2 Sn1.7 Cu/Au=315 Cu/Ag=52	Substrat	Cuivre Décalé *1.008

CuSn6Ag3Au1

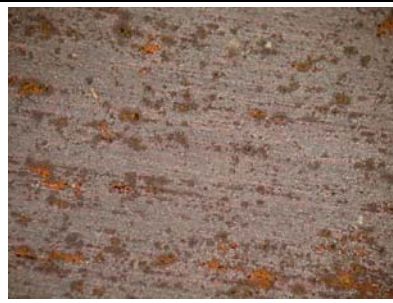
Recette :

Murakami :

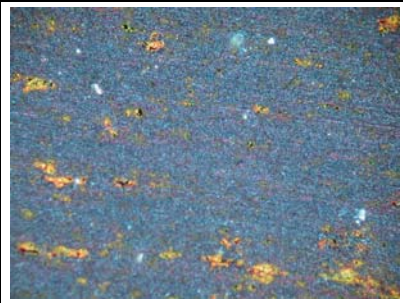
- 1,9 g acétate de cuivre
- 1,2 g sulfate de cuivre
- 0,1 g alun
- 1 l eau



MO BF

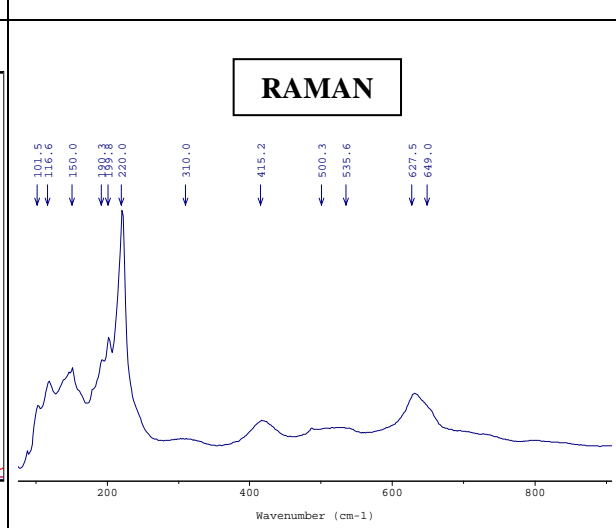
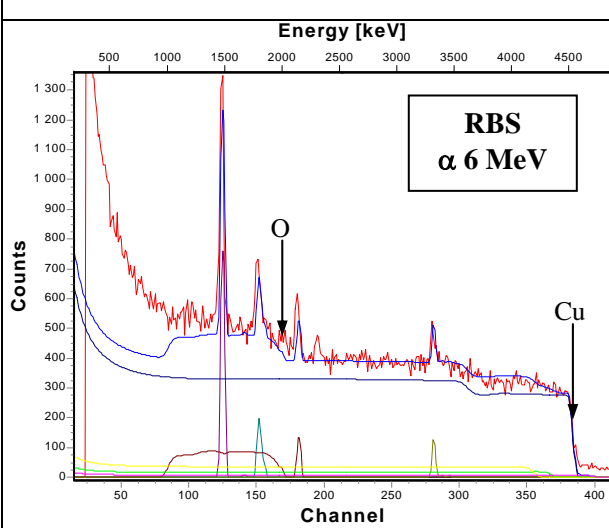
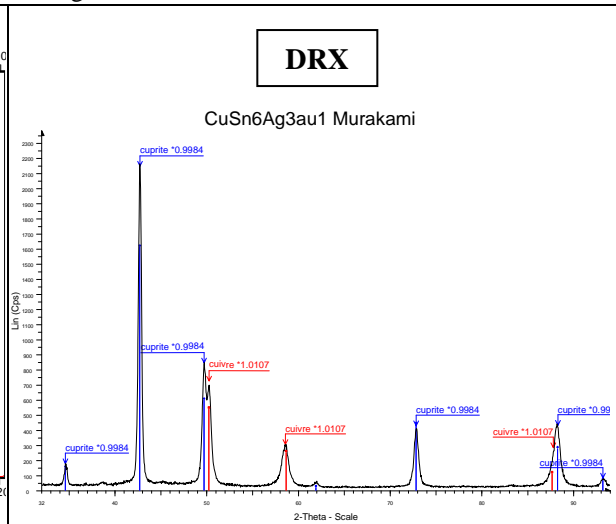
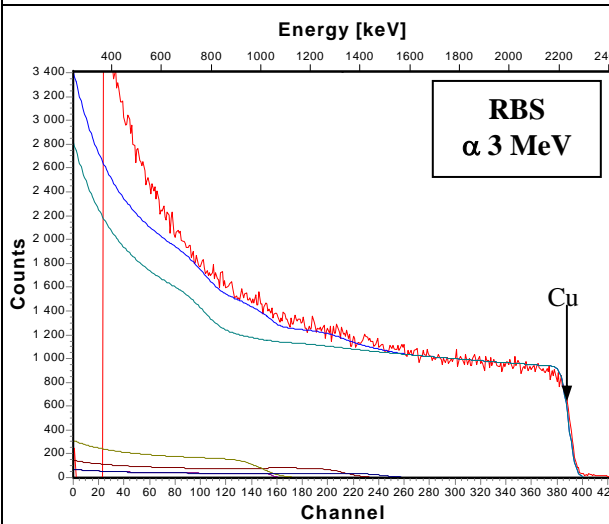


MO DF



MO PLA

PIXE: Cu89.1% Sn5.9% Ag3.2% Au0.9%



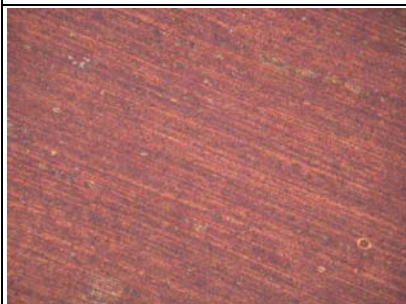
Description		
Composition Couche		
Cu67 O33 Ag0 Au0 rugosité	10000.10 ¹⁵ At/cm ²	Diffraction X
Cu67 Ag 1.8 Au0.3 O30.9 Cu/Au=220 Cu/Ag=36.6	3000.10 ¹⁵ At/cm ²	Cuprite Décalée *0.9984
Cu94.5 Ag1.8 Au0.3 Sn 3.4 Cu/Au=315 Cu/Ag=52	Substrat	Cuivre Décalé *1.0107

CuSn5Pb5

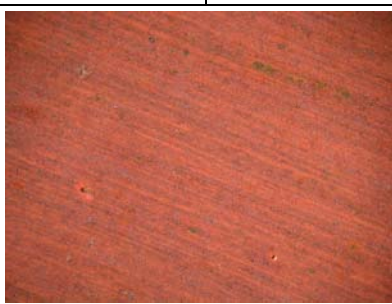
Recette :

Murakami :

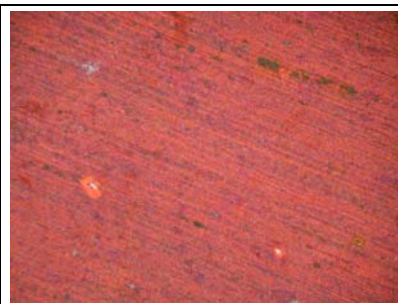
- 1,9 g acétate de cuivre
- 1,2 g sulfate de cuivre
- 0,1 g alun
- 1 l eau



MO BF

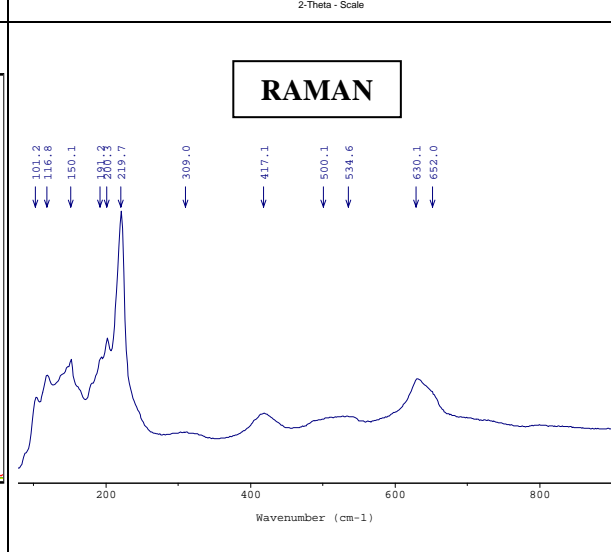
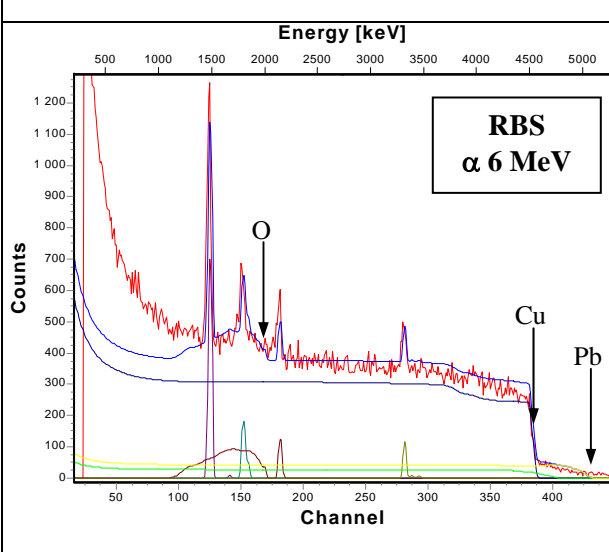
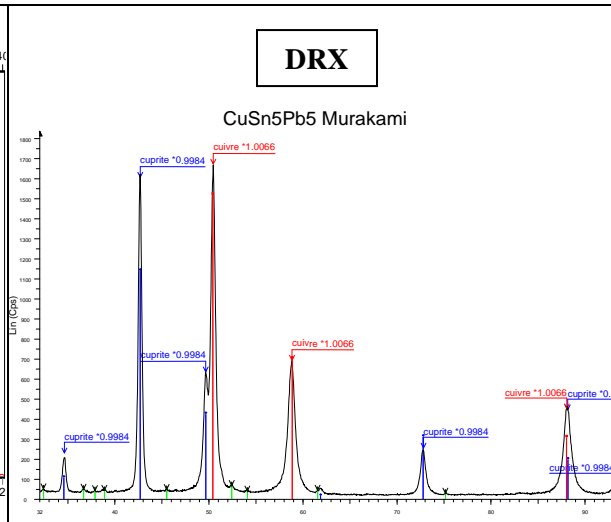
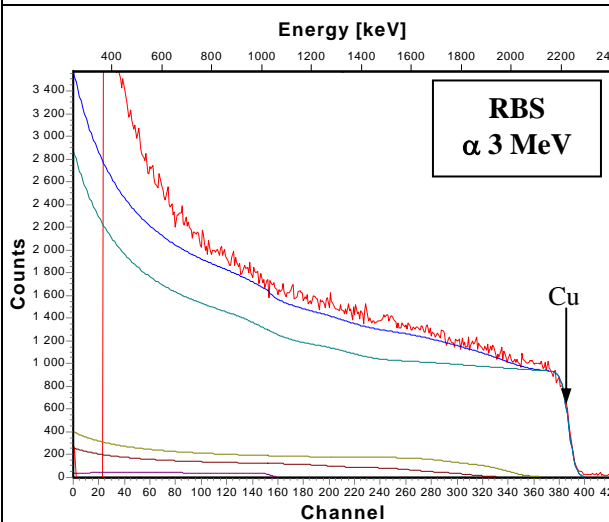


MO DF



MO PLA

PIXE: Cu94% Sn3.4% Pb2.6%



Description		
Composition Couche		
Cu66 O34 rugosité	6000.10 ¹⁵ At/cm ²	Diffraction X Cuprite Décalée *0.9984 Cuivre Décalé *1.0066
Cu68 Sn1 Pb1 O30	2000.10 ¹⁵ At/cm ²	
Cu78 Sn2 Pb1.5 O18.5	2500.10 ¹⁵ At/cm ²	
Cu95.6 Sn2.8 Pb1.6 Cu/Au=315 Cu/Ag=52	Substrat	