

Appendix



Table A.1: FSP parameters

No.	Rotational speed in rpm	Travel speed in mm/min	Feed rate in rev/min
C1	20	1600	0.0125
C2	20	1800	0.011
C3	20	2000	0.01
C4	40	1600	0.025
C5	40	1800	0.022
C6	40	2000	0.02
C7	60	1600	0.375
C8	60	1800	0.033
C9	60	2000	0.03
T1	20	1600	0.0125
T2	20	1800	0.011
T3	20	2000	0.01
T4	40	1600	0.025
T5	40	1800	0.022
T6	40	2000	0.02
T7	60	1600	0.375
T8	60	1800	0.033
T9	60	2000	0.03
S1	20	1600	0.0125
S2	20	1800	0.011
S3	20	2000	0.01
S4	40	1600	0.025
S5	40	1800	0.022
S6	40	2000	0.02
S7	60	1600	0.375
S8	60	1800	0.033
S9	60	2000	0.03
F1	20	1600	0.0125
F2	20	1800	0.011
F3	20	2000	0.01
F4	40	1600	0.025
F5	40	1800	0.022
F6	40	2000	0.02
F7	60	1600	0.375
F8	60	1800	0.033
F9	60	2000	0.03



Figure A.1: Cross sections of samples produced by the cylindrical profile

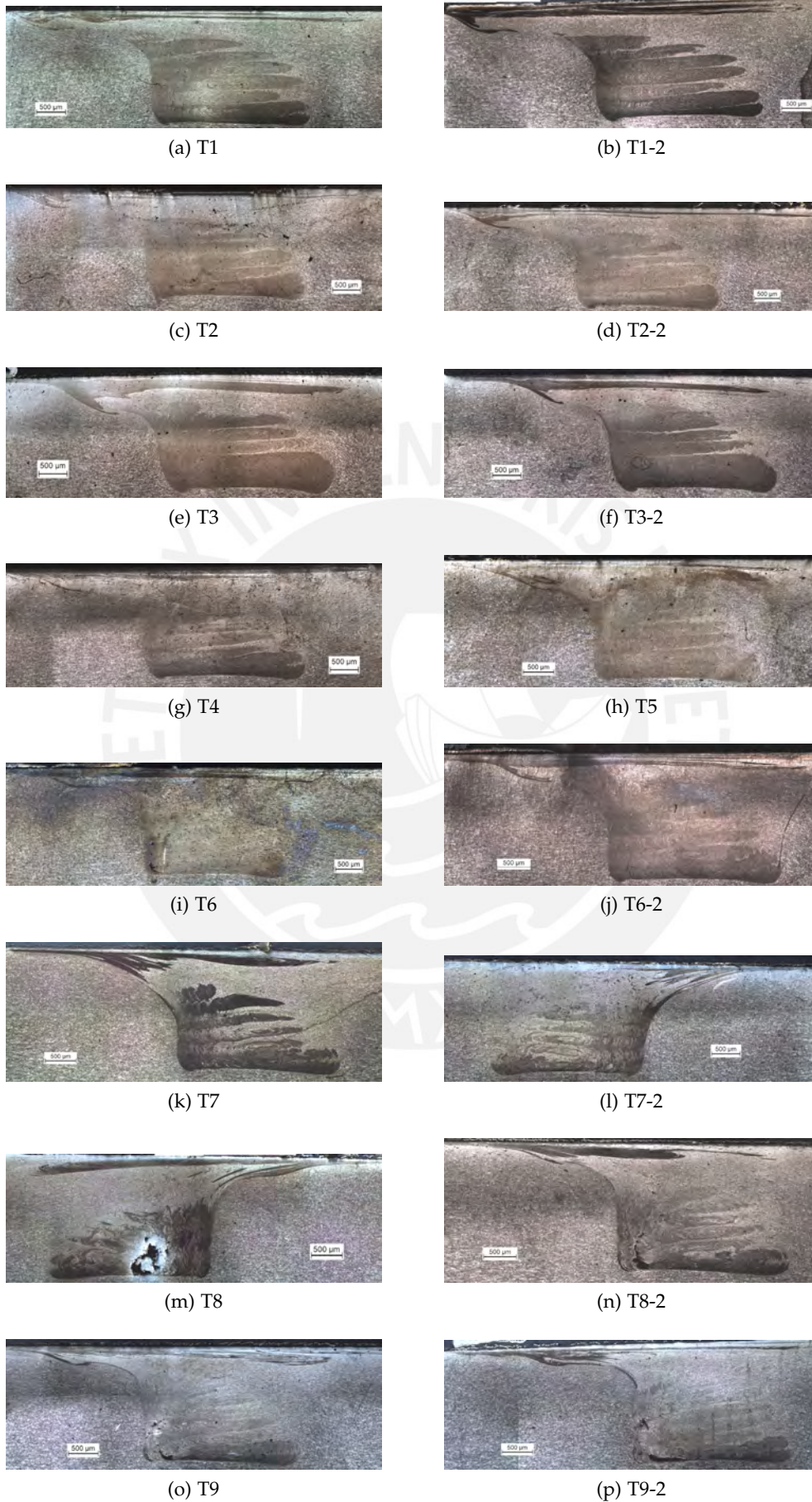


Figure A.2: Cross sections of samples produced by the triangular profile



(a) S1



(b) S1-2



(c) S2



(d) S2-2



(e) S3



(f) S3-2



(g) S4



(h) S4-2



(i) S5



(j) S5-2



(k) S6



(l) S6-2



(m) S7



(n) S7-2



(o) S8



(p) S9

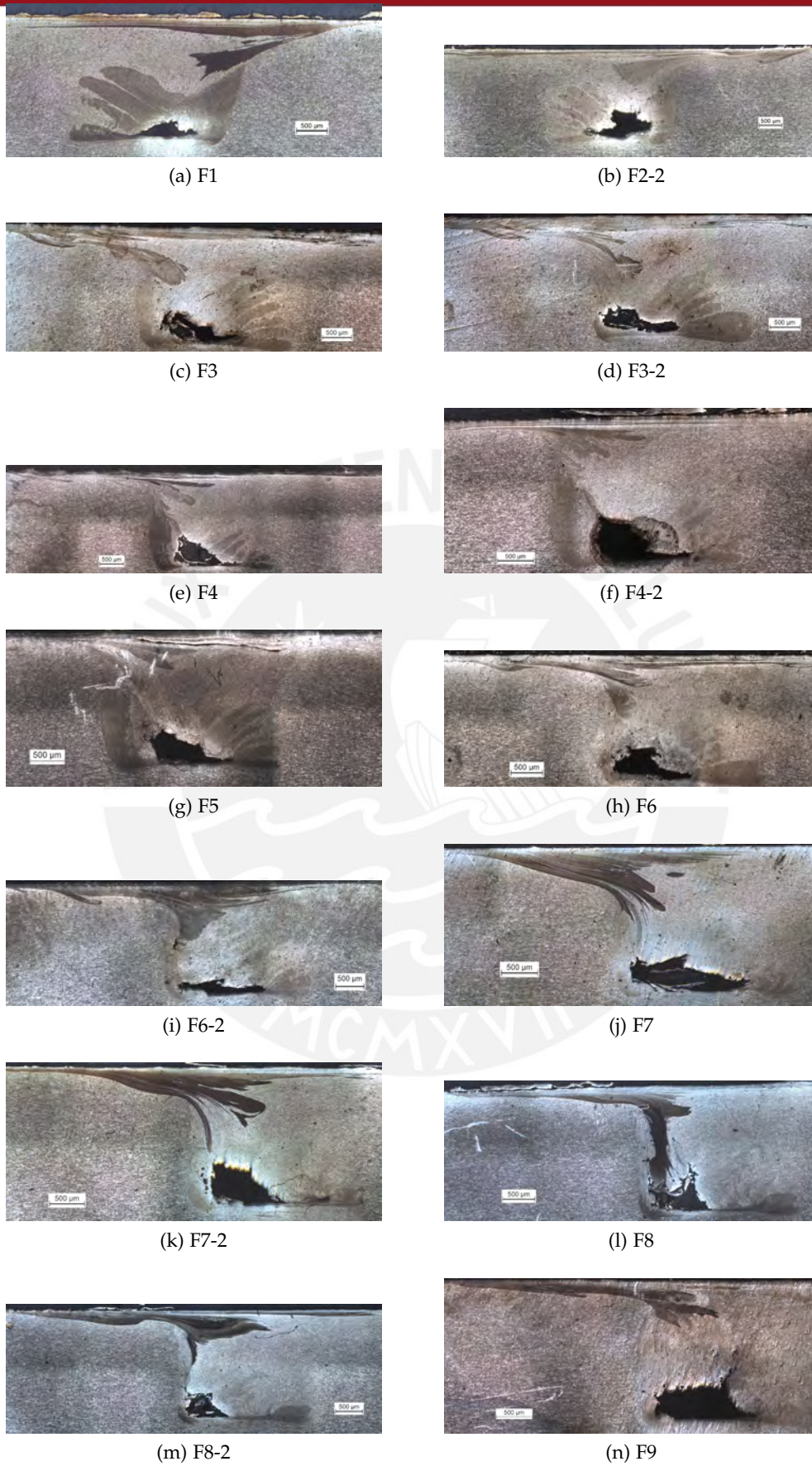
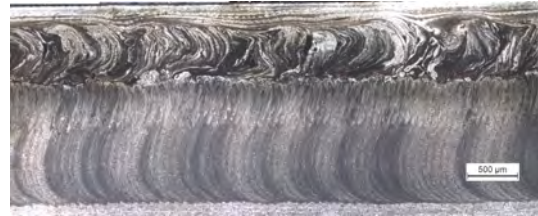


Figure A.4: Cross sections of samples produced by the fluted profile



(a) C1



(b) C2



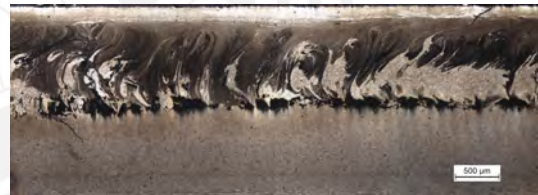
(c) C3



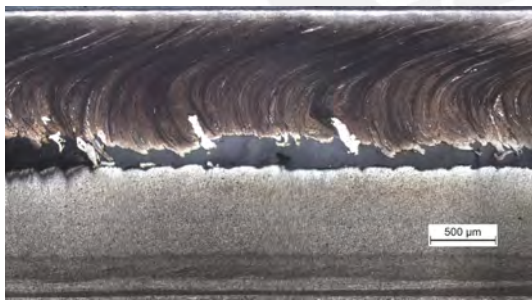
(d) C4



(e) C5



(f) C6



(g) C8



(h) C9

Figure A.5: Longitudinal sections of samples produced by the cylindrical profile

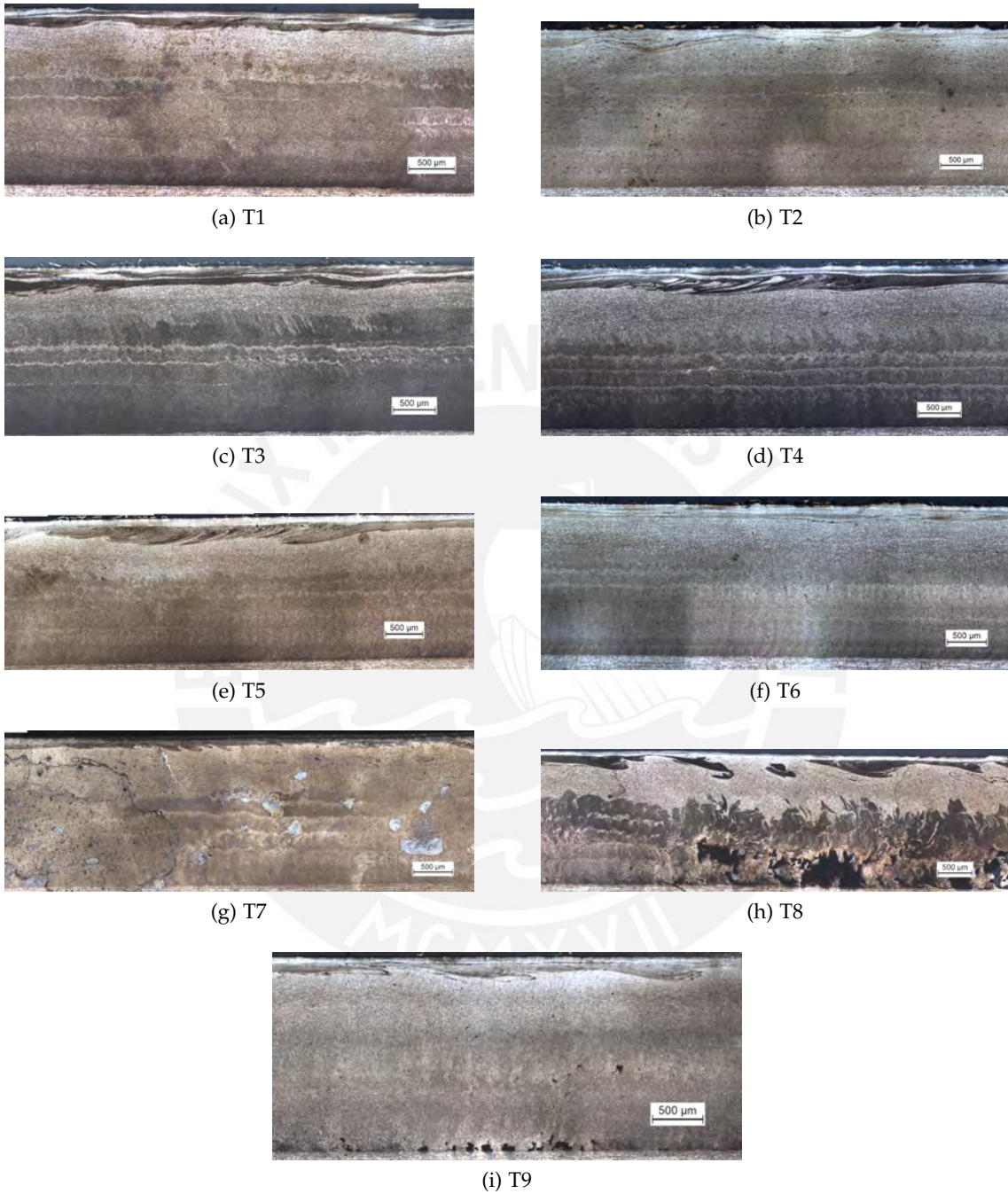
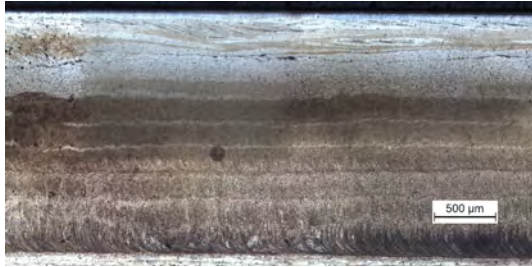


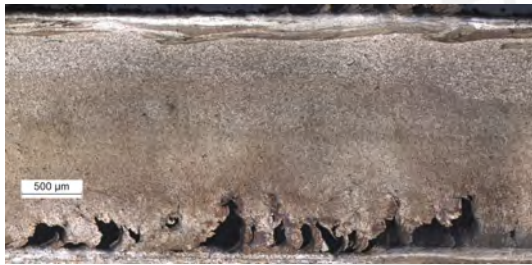
Figure A.6: Longitudinal sections of samples produced by the triangular profile



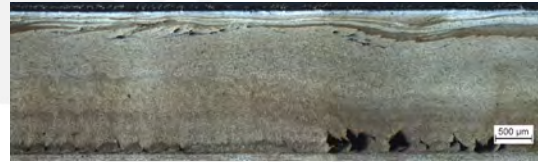
(a) S1



(b) S2



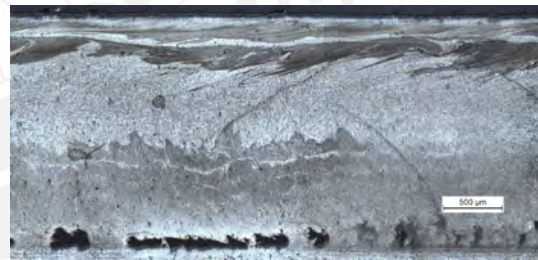
(c) S3



(d) S4



(e) S5



(f) S6



(g) S7

Figure A.7: Longitudinal sections of samples produced by the square profile

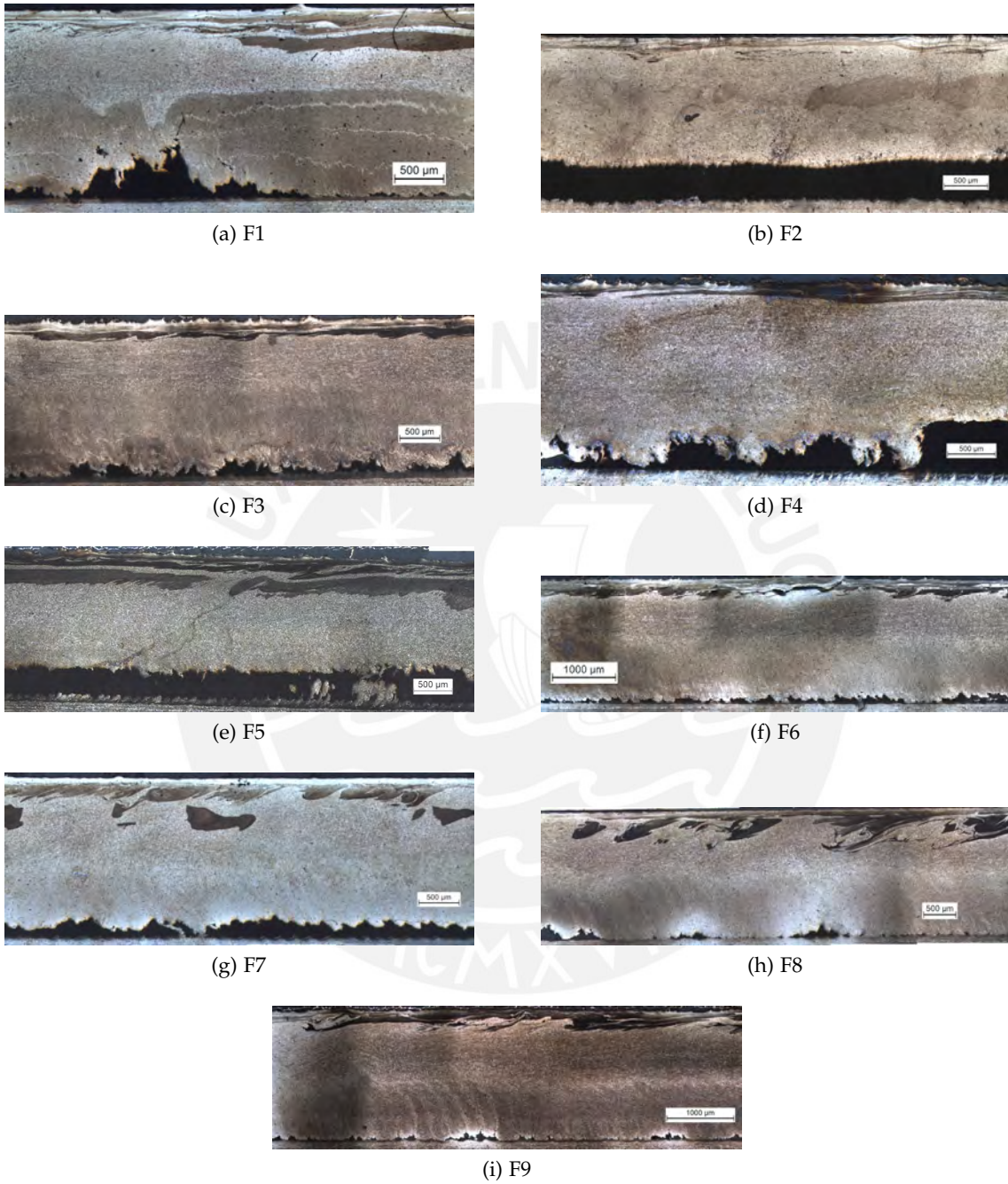
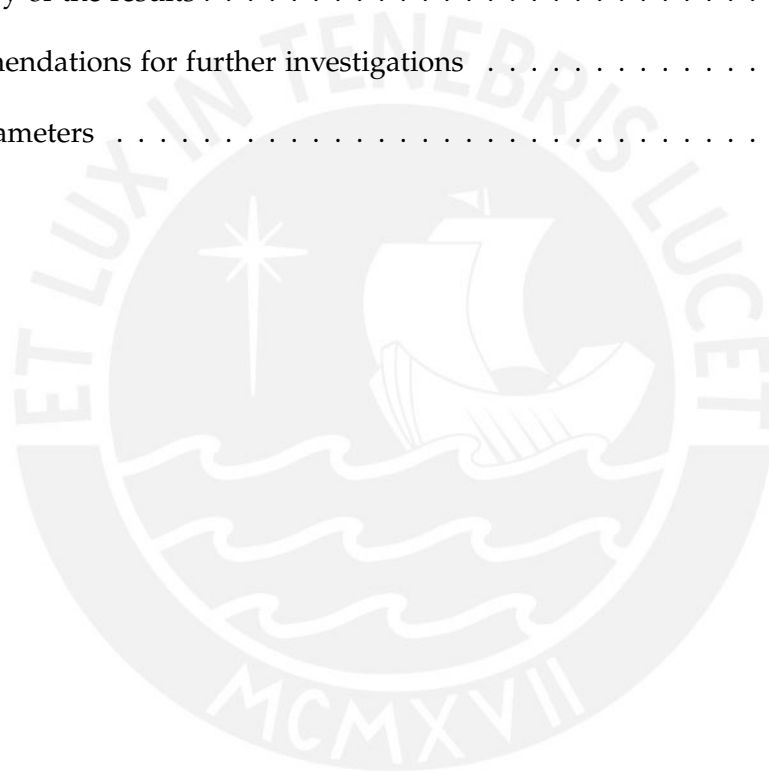


Figure A.8: Longitudinal sections of samples produced by the fluted profile

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Bibliography

- [Arb03] ARBEGAST, William J.: Friction Stir Joining: Characteristic Defects, South Dakota School of Mines and Technology, 2003
- [Arb08] ARBEGAST, William J.: A flow-partitioned deformation zone model for defect formation during friction stir welding. In: *Scripta Materialia* 58 (2008), Vol 5, 372 - 376. <http://dx.doi.org/10.1016/j.scriptamat.2007.10.031>. – DOI 10.1016/j.scriptamat.2007.10.031. – ISSN 1359–6462. – <ce:title>Viewpoint set no. 43 “Friction stir processing”</ce:title>
- [BCHS03] BARGEL, H.J. ; CARDINAL, P. ; HILBRANS, H. ; SCHULZE, G.: *Werkstoffkunde*. Springer, 2003 (VDI-Buch). <http://books.google.com.pe/books?id=pwHTAAACAAJ>. – ISBN 9783540401148
- [BLA10] BAKSHI, S R. ; LAHIRI, D ; AGARWAL, A: Carbon nanotube reinforced metal matrix composites; a review. In: *International Materials Reviews* 55 (2010), Vol 1, 41-64. <http://dx.doi.org/doi:10.1179/095066009X12572530170543>. – DOI doi:10.1179/095066009X12572530170543
- [BZH02] BAUGHMAN, Ray H. ; ZAKHIDOV, Anvar A. ; HEER, Walt A.: Carbon Nanotubes–the Route Toward Applications. In: *Science* 297 (2002), Vol 5582, 787-792. <http://dx.doi.org/10.1126/science.1060928>. – DOI 10.1126/science.1060928
- [CGJ+10] CHU, Ke ; GUO, Hong ; JIA, Chengchang ; YIN, Fazhang ; ZHANG, Ximin ; LIANG, Xuebing ; CHEN, Hui: Thermal Properties of Carbon Nanotube-Copper Composites for Thermal Management Applications. In: *Nanoscale Research Letters* 5 (2010), Vol 5, p 868–874. <http://dx.doi.org/10.1007/s11671-010-9577-2>. – DOI 10.1007/s11671-010-9577-2. – ISSN 1556–276X
- [Col99] COLLIGAN, K.: Material Flow Behavior during Friction Stir Welding of Aluminum. In: *Welding Journal* n/a (1999), p 229–237
- [EB08] ELANGOVA, K. ; BALASUBRAMANIAN, V.: Influences of tool pin profile and tool shoulder diameter on the formation of friction stir processing zone in AA6061 aluminium alloy. In: *Materials & Design* 29 (2008), Vol 2, 362 - 373. <http://dx.doi.org/10.1016/j.matdes.2007.01.030>. – DOI 10.1016/j.matdes.2007.01.030. – ISSN 0261–3069

- [EBV08] ELANGOVA, K. ; BALASUBRAMANIAN, V. ; VALLIAPPAN, M.: Influences of tool pin profile and axial force on the formation of friction stir processing zone in AA6061 aluminium alloy. In: *The International Journal of Advanced Manufacturing Technology* 38 (2008), 285-295. <http://dx.doi.org/10.1007/s00170-007-1100-2>. – DOI 10.1007/s00170-007-1100-2. – ISSN 0268-3768
- [GSM⁺02] GUERRA, M ; SCHMIDT, C ; McCLURE, J.C ; MURR, L.E ; NUNES, A.C: Flow patterns during friction stir welding. In: *Materials Characterization* 49 (2002), Vol 2, 95 - 101. [http://dx.doi.org/10.1016/S1044-5803\(02\)00362-5](http://dx.doi.org/10.1016/S1044-5803(02)00362-5). – DOI 10.1016/S1044-5803(02)00362-5. – ISSN 1044-5803
- [HBO⁺10] HIRASAWA, Shigeki ; BADARINARAYAN, Harsha ; OKAMOTO, Kazutaka ; TOMIMURA, Toshio ; KAWANAMI, Tsuyoshi: Analysis of effect of tool geometry on plastic flow during friction stir spot welding using particle method. In: *Journal of Materials Processing Technology* 210 (2010), Vol 11, 1455 - 1463. <http://dx.doi.org/10.1016/j.jmatprotec.2010.04.003>. – DOI 10.1016/j.jmatprotec.2010.04.003. – ISSN 0924-0136
- [IG12] IZADI, Hossein ; GERLICH, Adrian P.: Distribution and stability of carbon nanotubes during multi-pass friction stir processing of carbon nanotube/aluminum composites. In: *Carbon* 50 (2012), Vol 12, 4744 - 4749. <http://dx.doi.org/10.1016/j.carbon.2012.06.012>. – DOI 10.1016/j.carbon.2012.06.012. – ISSN 0008-6223
- [JYS⁺06] JOHANNES, Lucie B. ; YOWELL, Leonard L. ; SOSA, Edward ; AREPALLI, Sivaram ; ; MISHRA, Rajiv S.: Survivability of single-walled carbon nanotubes during friction stir processing. In: *Nanotechnology* 17 (2006), p 3081-3084
- [KK08] KUMAR, K. ; KAILAS, Satish V.: The role of friction stir welding tool on material flow and weld formation. In: *Materials Science and Engineering: A* 485 (2008), Vol 1-2, 367 - 374. <http://dx.doi.org/10.1016/j.msea.2007.08.013>. – DOI 10.1016/j.msea.2007.08.013. – ISSN 0921-5093
- [LKL⁺12] LIU, Qiang ; KE, Liming ; LIU, Fencheng ; HUANG, Chunping ; XING, Li: Microstructure and mechanical property of multi-walled carbon nanotubes reinforced aluminum matrix composites fabricated by friction stir processing. In: *Materials Design* 45 (2012), Vol 0, 343 - 348. <http://dx.doi.org/10.1016/j.matdes.2012.08.036>. – DOI 10.1016/j.matdes.2012.08.036. – ISSN 0261-3069
- [LMH10] LI, Hongjun ; MACKENZIE, Donald ; HAMILTON, Robert: Multi-Physics Simulation of Friction stir welding process. In: *Engineering Computations* 27 (2010), p 967-985. <http://dx.doi.org/10.1108/02644401011082980>. – DOI 10.1108/02644401011082980
- [LSG08] LIM, D.K. ; SHIBAYANAGI, T. ; GERLICH, A.P.: Synthesis of multi-walled CNT reinforced aluminium alloy composite via friction stir processing. In: *Materials*

- Science and Engineering: A* 507 (2008), Vol 1–2, 194 - 199. <http://dx.doi.org/10.1016/j.msea.2008.11.067>. – DOI 10.1016/j.msea.2008.11.067. – ISSN 0921–5093
- [Lui09] LUICK, Landon Q.: *FSP of SWNT to Increase the Thermal Conductivity of Aluminum*. South Dakota School of Mines and Technology, 2009
- [LXWM12] LIU, Z.Y. ; XIAO, B.L. ; WANG, W.G. ; MA, Z.Y.: Singly dispersed carbon nanotube/aluminum composites fabricated by powder metallurgy combined with friction stir processing. In: *Carbon* 50 (2012), Vol 5, 1843 - 1852. <http://dx.doi.org/10.1016/j.carbon.2011.12.034>. – DOI 10.1016/j.carbon.2011.12.034. – ISSN 0008–6223
- [Ma08] MA, Z.Y.: Friction Stir Processing Technology: A Review. In: *Metallurgical and Materials Transactions A* 39 (2008), 642-658. <http://dx.doi.org/10.1007/s11661-007-9459-0>. – ISSN 1073–5623. – 10.1007/s11661-007-9459-0
- [MM01] MISHRA, Rajiv S. ; MAHONEY, Murray W.: Friction Stir Processing: A New Grain Refinement Technique to Achieve High Strain Rate Superplasticity in Commercial Alloys. In: *Materials Science Forum* 357-359 (2001), p 507–514. <http://dx.doi.org/10.4028/www.scientific.net/MSF.357-359.507>. – DOI 10.4028/www.scientific.net/MSF.357–359.507
- [MM05] MISHRA, R.S. ; MA, Z.Y.: Friction stir welding and processing. In: *Materials Science and Engineering: R: Reports* 50 (2005), Vol 1–2, 1 - 78. <http://dx.doi.org/10.1016/j.mser.2005.07.001>. – DOI 10.1016/j.mser.2005.07.001. – ISSN 0927–796X
- [MM07] MISHRA, Rajiv S. ; MAHONEY, Murray W. ; MISHRA, Rajiv S. (Hrsg.) ; MAHONEY, Murray W. (Hrsg.): *Friction Stir Welding and Processing*. ASM International, 2007. – 1–352 S. <http://dx.doi.org/10.1361/fswp2007p001>. <http://dx.doi.org/10.1361/fswp2007p001>
- [PT97] P.L. THREADGILL, March 1. TWI Bull. B. TWI Bull.: TWI Bulletin. In: *TWI Bulletin* n/a (1997), p n/a
- [RDBD10] RAI, R ; DE, A ; BHADOSHIA, H K D H. ; DEBROY, T: Review: friction stir welding tools. In: *Science and Technology of Welding & Joining* 16 (2010), Vol 4, 325-342. <http://dx.doi.org/doi:10.1179/1362171811Y.0000000023>. – DOI doi:10.1179/1362171811Y.0000000023
- [SHBK08] SCHULZE, G. ; HILBRANS, H. ; BARGEL, H.-J. ; KRÜGER, O. ; SCHULZE, G. (Hrsg.) ; BARGEL, H.-J. (Hrsg.): *Werkstoffkunde*. 10. bearbeitete Auflage. Berlin, Heidelberg, New York : Springer, 2008. – ISBN 9783540792963
- [TCZC04] TANG, Yongbing ; CONG, Hongtao ; ZHONG, Rong ; CHENG, Hui-Ming: Thermal expansion of a composite of single-walled carbon nanotubes and nanocrystalline aluminum. In: *Carbon* 42 (2004), p 3260–3262. <http://dx.doi.org/10.1016/j.carbon.2004.07.024>. – DOI 10.1016/j.carbon.2004.07.024

- [THG⁺12] TUTUNCHILAR, S. ; HAGHPANAHI, M. ; GIVI, M.K. B. ; ASADI, P. ; BAHEMMAT, P.: Simulation of material flow in friction stir processing of a cast Al-Si alloy. In: *Materials & Design* 40 (2012), Vol 0, 415 - 426. <http://dx.doi.org/10.1016/j.matdes.2012.04.001>. – DOI 10.1016/j.matdes.2012.04.001. – ISSN 0261-3069
- [Tho03] THOMAS, W.M.: Friction Stir Welding - Recent Developments. In: *Materials Science Forum THERMEC'2003* (2003), p 229-236. <http://dx.doi.org/10.4028/www.scientific.net/MSF.426-432.229>. – DOI 10.4028/www.scientific.net/MSF.426-432.229
- [TJW03] THOMAS, W.M. ; JOHNSON, K.I. ; WIESNER, C.S.: Friction Stir Welding – Recent Developments in Tool and Process Technologies. In: *Advanced Engineering Materials* 5 (2003), Vol 7, p 485-490. <http://dx.doi.org/10.1002/adem.200300355>. – DOI 10.1002/adem.200300355. – ISSN 1527-2648
- [TNN⁺96] THOMAS, W. M. ; NICHOLAS, E. D. ; NEEDHAM, J. C. ; TEMPLE-SMITH, P. ; KALLEE, S. W. K. W. ; DAWES, C. J. (: *Friction Stir Welding, UK Patent Application GB 2 306 366 A*. 1996
- [TNS01] THOMAS, W.M. ; NICHOLAS, E.D. ; SMITH, S.D.: *Friction Stir Welding-Tool Developments*. Aluminum 2001, Proceedings of the TMS 2001 Aluminum Automotive and Joining Sessions, 2001
- [TSNF03] THOMAS, W. M. ; STAINES, D. G. ; NORRIS, I. M. ; FRIAS, R. de: Friction stir welding tools and developments. In: *Welding in the world* 47 (2003), Vol 11-12, p 10-17
- [ZCLW12] ZHANG, Y N. ; CAO, X ; LAROSE, S ; WANJARA, P: Review of tools for friction stir welding and processing. In: *Canadian Metallurgical Quarterly* 51 (2012), Vol 3, 250-261. <http://dx.doi.org/doi:10.1179/1879139512Y.0000000015>. – DOI doi:10.1179/1879139512Y.0000000015
- [ZLQW06] ZHAO, Y.-H. ; LIN, S.-B. ; QU, F.-X. ; WU, L.: Influence of pin geometry on material flow in friction stir welding process. In: *Materials Science and Technology* 22 (2006), Vol 1, 45-50. <http://dx.doi.org/doi:10.1179/174328406X78424>. – DOI doi:10.1179/174328406X78424
- [ZZ08] ZHANG, Z. ; ZHANG, H.W.: A fully coupled thermo-mechanical model of friction stir welding. In: *The International Journal of Advanced Manufacturing Technology* 37 (2008), 279-293. <http://dx.doi.org/10.1007/s00170-007-0971-6>. – DOI 10.1007/s00170-007-0971-6. – ISSN 0268-3768